

**REMEDIAL ACTION CONTRACT 2
FOR REMEDIAL INVESTIGATION/FEASIBILITY STUDY (RI/FS)
IN REGION 5**

ATTACHMENT A

**FIELD SAMPLING PLAN
USS LEAD SUPERFUND SITE
EAST CHICAGO, LAKE COUNTY, INDIANA**

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Attachment

A	STANDARD OPERATING PROCEDURES
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ACRONYMS AND ABBREVIATIONS

μm	Micrometer
°C	Degree Celsius
ARCO	Anaconda Copper Company
ASTM	American Society for Testing and Materials
bgs	Below ground surface
CAMU	Corrective Action Management Unit
CLP	Contract Laboratory Program
COC	Chain of custody
CPR	Cardiopulmonary resuscitation
CRL	Central Regional Laboratory
dpi	Dots per inch
DOT	U.S. Department of Transportation
Dupont	E.I. duPont de Nemours Company
FSP	Field sampling plan
HASP	Health and safety plan
HDPE	High-density polyethylene
Hg	Mercury
ID	Identification
IDEM	Indiana Department of Environmental Management
IDW	Investigation-derived waste
ISBH	Indiana State Board of Health
mL	Milliliter
MS	Matrix spike
MSD	Matrix spike duplicate
NA	Not applicable
NPDES	National Pollutant Discharge Elimination System
NPL	National Priorities List
PCB	Polychlorinated biphenyl
PPE	Personal protective equipment
ppm	Parts per million
PTFE	Polytetrafluoroethylene

QA	Quality assurance
QAPP	Quality assurance project plan
QC	Quality control
RAC	Remedial action contract
RCRA	Resource Conservation and Recovery Act
ROD	Record of Decision
SAP	Sampling and analysis plan
SMO	Sample Management Office
SOP	Standard operating procedure
SOW	Statement of work
SVOC	Semivolatile organic compound
TAL	Target Analyte List
TSCA	Toxic Substance and Control Act
USCS	Unified Soil Classification System
USDA	U.S. Department of Agriculture
U.S. EPA	U.S. Environmental Protection Agency
USS Lead	U.S. Smelter and Lead Refinery, Inc.
VOC	Volatile organic compound
WA	Work assignment
XRF	X-ray fluorescence

A1.0 INTRODUCTION

Under the U.S. Environmental Protection Agency (U.S. EPA) Remedial Action Contract (RAC) 2 for Region 5, Contract No. EP-S5-06-02, Work Assignment (WA) No. 054-RICO-053J, SulTRAC has prepared this field sampling plan (FSP) as part of the sampling and analysis plan (SAP) for the USS Lead Superfund (USS Lead) Site in Lake County, Indiana (see [Figure A-1](#)). The SAP consists of this FSP (Appendix A) and the quality assurance project plan (QAPP) ([Appendix B](#)), which are among the site-specific plans to be prepared under the WA in accordance with Task 1 of the U.S. EPA statement of work (SOW) (EPA 2009). The QAPP discusses quality assurance (QA) and quality control (QC) protocols associated with sampling and analysis activities at the USS Lead Site.

This FSP describes sampling activities SulTRAC will perform during the remedial investigation at the USS Lead site. As outlined in the USS Lead site work plan ([SulTRAC 2009a](#)), the scope of this FSP was developed based on interaction with the U.S. EPA and review of site-related documents. FSP field investigation activities will focus on the following activities and areas:

- Determine the lateral and vertical extent of lead-contaminated soils at residences, schools, parks, vacant lots, and other areas where children may come into contact with contaminated soil
- Determine whether other contaminants are associated with lead-contaminated soils
- Determine whether the lead-contaminated soil is associated with size fractions that pass through an ASTM Standard No. 60 sieve (sieve openings 250 micrometers [μm], or 0.009 inches)
- Determine whether the lead-contaminated soil requires disposal as characteristic hazardous waste
- Provide a good basis for estimating the number of homes in the study area that will require remediation
- Evaluate contamination in an on-site wetland and determine whether the contamination merits a remedial response

This FSP discusses

- the site background ([Section A2.0](#)),
- project objectives ([Section A3.0](#)),
- field sampling activities ([Section A4.0](#)),
- field sampling procedures ([Section A5.0](#)),

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- laboratory analytical methods ([Section A6.0](#)),
 - decontamination procedures ([Section A7.0](#)),
 - sample handling procedures ([Section A8.0](#)),
 - disposal of investigation-derived waste (IDW) ([Section A9.0](#)),
 - health and safety procedures ([Section A10.0](#)),
 - QA/QC requirements ([Section A11.0](#)).
 - [Section A12.0](#) lists references used to prepare this FSP.

Tables are included in the body of the text. [Figures](#) are provided after Section A12.0. Standard operating procedures (SOP) are provided in [Attachment A](#).

A2.0 SITE BACKGROUND

This section discusses the site description ([Section A2.1](#)), site history ([Section A2.2](#)), previous investigations ([Section A2.3](#)), and current status ([Section A2.4](#)).

A2.1 SITE DESCRIPTION

The former site of the U.S Smelter and Lead Refinery, Inc. (USS Lead) operation is located on a 79-acre parcel of land in East Chicago, Indiana. The area is primarily industrial with a nearby residential area to the north of the site. The old plant location is bordered by the Indiana Harbor Belt Railroad to the north; the east branch of the Grand Calumet River to the south, Kennedy Avenue to the east, and the Indiana Harbor Canal to the west.

A2.2 SITE HISTORY

From 1906 to 1920, the company added a primary lead smelter to its operation. USS Lead converted to secondary smelting in 1973, recovering lead from scrap metal and old automobile batteries. All operations were discontinued in 1985. Two primary waste materials were generated as a result of the smelting operations: 1) blast furnace slag and 2) lead-containing dust emitted by the blast furnace stack. Blast-furnace slag was stockpiled south of the plant building and spread over an adjoining 21 acres of wetlands once per year. The lead-containing dust was originally trapped in bag filters and stored in a 3- to 5-acre area for future recycling ([EPA 2009](#)).

In 1975 and 1985, USS Lead received a National Pollutant Discharge Elimination System (NPDES) permit to discharge furnace cooling water and storm water runoff to the Grand Calumet River. According to the Indiana Department of Environmental Management (IDEM), discharge exceeded permit levels for several compounds. In the 1980s, several state and federal enforcement actions were taken against the company. In September 1985, the Indiana State Board of Health (ISBH) found USS Lead in violation of State law because lead particles were found downwind of the site. Approximately four million people draw drinking water from intakes primarily into Lake Michigan, which is 15 miles downstream of where hazardous substances from the site enter surface water. Seventy-five hundred people work or attend school within two miles of the site ([EPA 2009](#)).

A2.3 PREVIOUS SITE INVESTIGATIONS AND REMOVAL ACTIONS

Since 1985, U.S. EPA Resource Conservation and Recovery Act (RCRA) Corrective Action has overseen the remediation and management of lead-contaminated soils within the boundaries of the former USS Lead facility. The remediation of the facility included the placement of some of the most contaminated sediments and soils in a Corrective Action Management Unit (CAMU). The remediation included a 2-acre section of the on-site wetlands, where wetland soils and sediments were contaminated with lead at concentrations in excess of 10,000 milligrams per kilogram (mg/kg) (DAI Environmental Inc. 2004).

The residential area north of the site includes about 1,000 homes, a few parks, schools, and public buildings, and has been sampled several times by several different entities. The residential area itself has been sampled multiple times by various different groups as follows: EPA in 1985; Entact in 1999; EPA/IDEM in 2002; EPA RCRA in 2003; and EPA in 2006.

In 2003, U.S. EPA sampled soils in the residential area north of USS Lead as a part of the RCRA Corrective Action investigation. These sampling results showed some residential yards to have high levels of lead contamination. Most of the yards with the highest lead sampling results were in the southern region of the residential area. In 2005, U.S. EPA RCRA Corrective Action referred USS Lead to Superfund for the cleanup of the residential portion of the site. In 2006, RCRA Corrective Action amended the referral to include the wetlands, as a part of the referred areas.

In April 2006, U.S. EPA Superfund re-sampled the yards at 14 properties. The analysis of those samples confirmed that the yards for at least 12 homes had lead contamination levels above 1200 parts per million (ppm) (the Tier 1 level used in the evaluation of residential yards with lead contamination).

In 2008, the Superfund removal program remediated yards at 13 properties that were above Tier 1 levels.

A2.4 CURRENT SITE STATUS

The USS Lead refinery is currently inactive. The site consists of a residential area and a wetlands area (Figure A-2). Sampling in the wetlands area will be addressed separately and is not discussed further in this FSP. For the purposes of this investigation, the residential portion of the study area consists of the area bounded by the Indiana Harbor Canal to the west, Chicago Avenue to the north, Parrish Avenue to the east (Figure A-3). The southern boundary of the residential portion of the study area is defined by East 151st Street from the canal to Huish Drive, the southernmost railroad tracks from Huish Drive to Grasselli Street, and East 149th Place from Grasselli Street to Parrish Avenue (Figure A-2). The area defined by

these boundaries contains 15 residential blocks (about 390 homes) east of the railroad tracks; 14 residential blocks (about 375 homes) west of the railroad tracks; one residential block and four half-blocks of residences (about 75 homes) on the west side of McCook Avenue; and a large public housing complex with 96 individual dwellings and 2 multistory apartment complexes west of McCook Avenue. In total, the residential study area contains about 940 homes.

The enforcement team has not identified any potentially responsible parties with the ability to finance the proposed response actions. U.S. EPA has focused its investigations on three legal persons who are potentially responsible for the contamination. These persons are: U.S. Smelter and Lead Refinery, Inc. (USS Lead); Atlantic Richfield Company, successor in interest to Anaconda Copper Company (ARCO); and E.I. duPont de Nemours Company (DuPont). U.S. EPA has concluded that (1) USS Lead has no ability to pay for a cleanup; (2) DuPont has an ability to pay for a removal action, but U.S. EPA may not be able to prove that DuPont disposed of hazardous substances at the site; and (3) ARCO has a limited ability to pay for a cleanup and U.S. EPA may not be able to prove that hazardous substances were released during the period of time in which ARCO's predecessor in interest owned the property. Under these circumstances, this investigation and subsequent remedial actions will be a fund-financed removal action with few prospects for recovering incurred costs.

A3.0 PROJECT OBJECTIVES

The purpose of this FSP is to describe the approach that will be used to conduct the remedial investigation at the USS Lead Site. The objectives of the investigation are to:

- Determine the lateral and vertical extent of lead-contaminated soils at residences, schools, parks, vacant lots, and other areas where children may come into contact with contaminated soil;
- Determine whether other contaminants are associated with lead-contaminated soils;
- Determine whether the lead-contaminated soil is associated with size fractions that pass through an ASTM Standard No. 60 sieve (sieve openings of 250 μm , 0.009 inches);
- Determine whether the lead-contaminated soil requires disposal as characteristic hazardous waste; and
- Provide a good basis for estimating the number of homes in the study area that will require remediation.

The investigation will be conducted in two phases. The Phase I investigation will conduct sampling in the residential area on a widely spaced sampling grid to further refine the area that requires remedial action, and to identify whether lead contamination is associated with specific geographic areas, particle sizes, or other contaminants. The second phase of the investigation will be based on the results of the first phase of investigation, and will identify individual residences that require remediation. Investigation of the wetlands area will be pursued independently from the investigation of the residential area, and will not be addressed further in this FSP.

All SulTRAC field activities will be conducted in accordance with the U.S. EPA-approved, site-specific QAPP ([Appendix B](#)) and SulTRAC SOPs (see [Attachment A](#)). Where this FSP differs from the SOPs, the FSP's site-specific procedures will take precedence.

After the Phase I investigation is complete, a data evaluation summary report will be prepared to document the results. The results of the Phase I investigation will be used to identify data gaps. The data gaps will be further addressed during the Phase II site investigation. SulTRAC and U.S. EPA will determine whether risk assessments will be prepared to evaluate the actual or potential risks to human health and the environment from the USS Lead site.

A4.0 FIELD SAMPLING APPROACH

The field sampling approach discussed in this section pertains to the SulTRAC site investigation, which will be performed in two phases. [Section A5.0](#) provides a detailed discussion of sample collection procedures.

As discussed with the U.S. EPA and outlined in the work plan ([SulTRAC 2009a](#)), SulTRAC will conduct site investigation activities in two phases. Phase I of the investigation will include extensive X-ray fluorescence (XRF) screening investigation (approximately 1,200 samples) at residential properties distributed in a widely-spaced grid pattern across the entire residential area. Further, the Phase I Investigation will collect additional samples from the same locations to resolve potential issues identified in the Superfund Lead-Contaminated Residential Sites Handbook ([US EPA 2003](#)). The second phase of the investigation will use the results of the Phase I activities to identify a more focused investigation area from which to collect samples, using a more closely-spaced sampling pattern.

EPA's Work Assignment Manager provided a package of 132 residential access agreements from March and April 2006 ([Figure A-3](#)). Additional residential access agreements will be required before initiating Phase I of the investigation.

SulTRAC will submit 20% of the XRF-screened soil samples to a Contract Laboratory Program (CLP) laboratory for metals analysis to determine correlation values between the XRF screenings and CLP analytical results. The objective of subjecting 20% of the XRF-screened samples to CLP metals analysis is to develop a site-specific correlation between field XRF metals concentrations and CLP laboratory metals concentrations. SulTRAC will perform a regression analysis on the resulting data and will use the regression to develop a site-specific correction factor for XRF data. CLP laboratory metals concentrations will be used for samples and analytes where both XRF and CLP laboratory data are available. Samples with only XRF concentration data will be corrected using the regression curve to derive a more accurate and precise concentration. Resulting data will be used to make decisions regarding remedial actions at properties with lead concentrations below 400 mg/kg or above 1,200 mg/kg. The Superfund Lead-Contaminated Residential Sites Handbook ([US EPA 2003](#)) suggests that 20% laboratory confirmation samples will provide sufficient data to evaluate the accuracy and precision of the XRF data.

SulTRAC will collect a variety of other samples to address other potential potential issues identified in the Superfund Lead-Contaminated Residential Sites Handbook ([US EPA 2003](#)) as described below. SulTRAC will submit one sample each from approximately 5% of the properties to a CLP laboratory for

sieve analysis to determine whether contamination is associated with the finer grain-size fraction, as recommended by the Superfund Lead-Contaminated Residential Sites Handbook ([US EPA 2003](#)). One sample each from approximately 10% of the properties sampled will be analyzed for a full scan of contaminants, including volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs), polychlorinated biphenyl (PCBs), and pesticides. Where possible, the samples for full-scan and sieve analysis samples will be collected from the same properties and depths. However, the VOC samples will not be collected from the uppermost 6 inches below the ground surface (bgs), so the VOC samples may be collected from different depth intervals. VOC samples will not be composited due to the tendency of VOCs to volatilize. Instead, a single VOC sample will be collected from the component of the composite sample that exhibits the most visual or olfactory evidence of contamination. If no contamination is evident, a single sample will be randomly selected for VOC analysis. [Table A-1](#) provides a sampling summary.

The following sections discuss the sampling approaches to be used for Phase I of the remedial investigation. After completion of the Phase I investigation, SulTRAC will amend the SAP and associated FSP and QAPP to include additional information related to the Phase II investigation.

A4.1 PHASE 1 SITE INVESTIGATION

The Phase I site investigation will include the collection of XRF soil screening and samples on a widely-spaced grid pattern throughout the residential study area. SulTRAC has used the city blocks as a basis to delineate the lateral extent of lead contamination. SulTRAC will collect samples from properties on each side of each block, for a total of approximately 3 properties per block (two on one side and one on the other). The field team will attempt to distribute the samples to provide even coverage of the study area; properties to be sampled will be determined in the field based on site access. The residential area includes approximately 30 residential blocks, so a total of 90 properties will be sampled during the first phase of the investigation. An additional 20 samples will be collected in the area between McCook Avenue and the Indiana Harbor Canal, and 5 additional samples will be collected in schools and parks, for a total of 115 properties evaluated during the initial phase of the investigation.

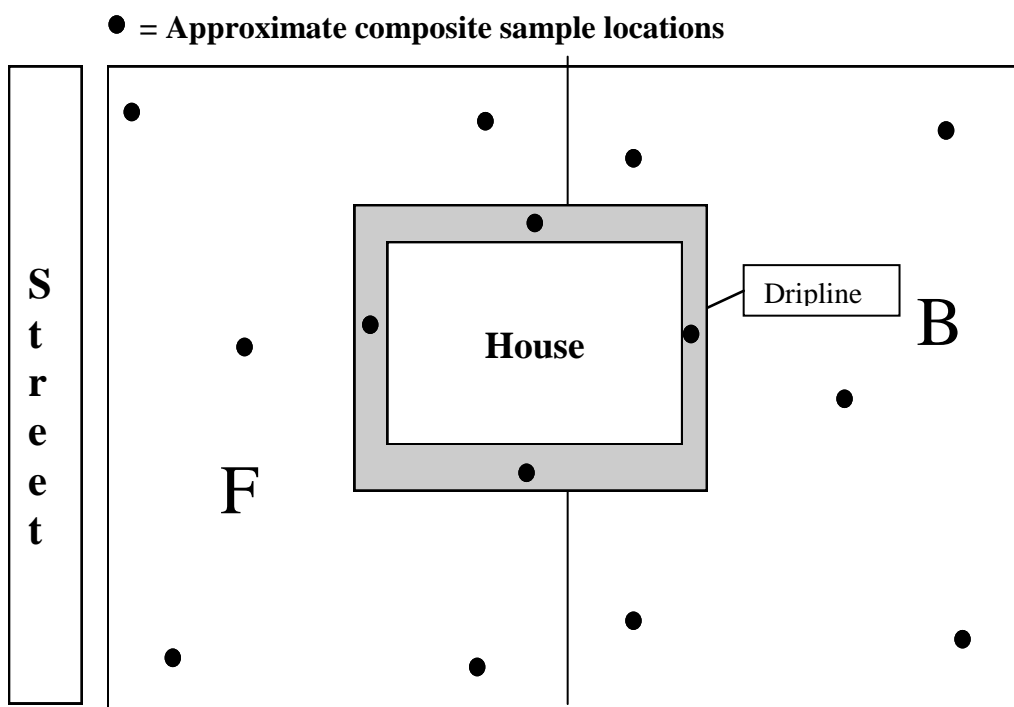
A4.1.1 Soil Screening and Sampling Approach

SulTRAC will mobilize to the site to conduct an XRF soil screening investigation for metals. The goal of the screening is to identify residential properties above the Toxic Substance and Control Act (TSCA) Section 403 soil lead hazard level of 400 ppm.

A4.1.1.1 Residential Properties

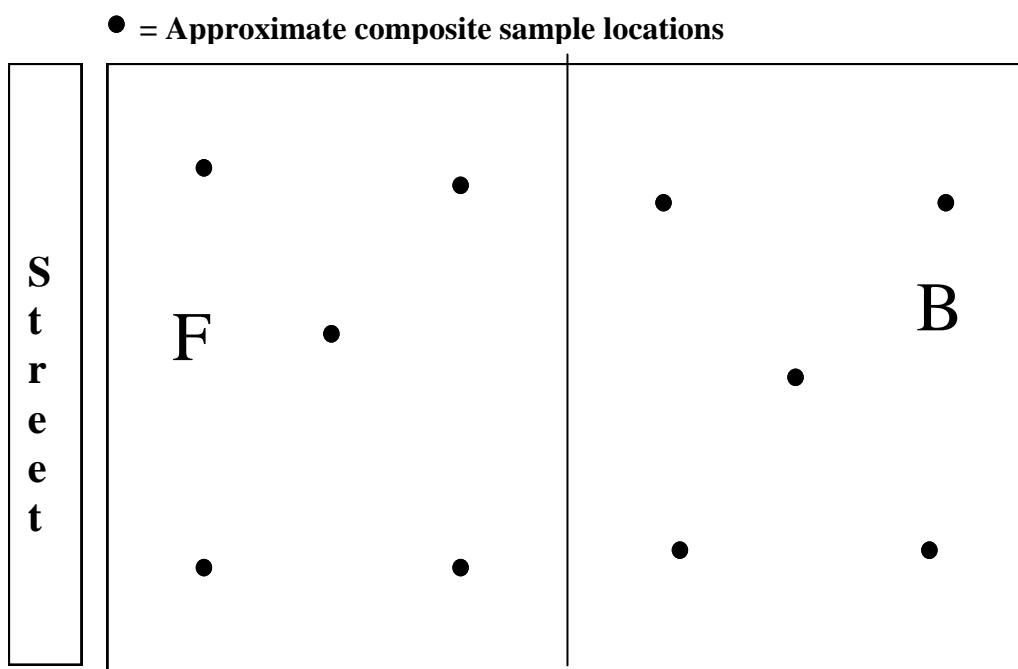
Residential properties with a structure on the property will be divided into front and back yards, and a 5-point composite sample will be collected from each front yard and each back yard. If there are side yards, the 5-point composite will include locations from the side yards. Four depth-discrete 5-point composite samples will be collected from each yard, including 5-point composite samples from 0-6 inches, 6-12 inches, 12-18 inches and 18-24 inches bgs, in an X-shaped pattern, with one sample from each end point of the X and one sample from the center. One four-point composite surface sample will be collected from 0-6 inches from the drip line area surrounding the house from each residential property. If the property has gutters, surface samples collected from each gutter outfall location will be composited. Typically a total of 9 samples will be collected at each residential property

SulTRAC will collect grab samples from each of four sample depths (0-6 inches, 6-12 inches, 12-18 inches, and 18-24 inches bgs) from a single location in the approximate center of the each vegetable or flower garden areas. If there are multiple gardens located on a property, each garden area will be sampled separately. SulTRAC will also collect grab samples from each of four sample depths (0-6 inches, 6-12 inches, 12-18 inches, and 18-24 inches bgs) from any play areas (e.g., swing sets) located on a property.



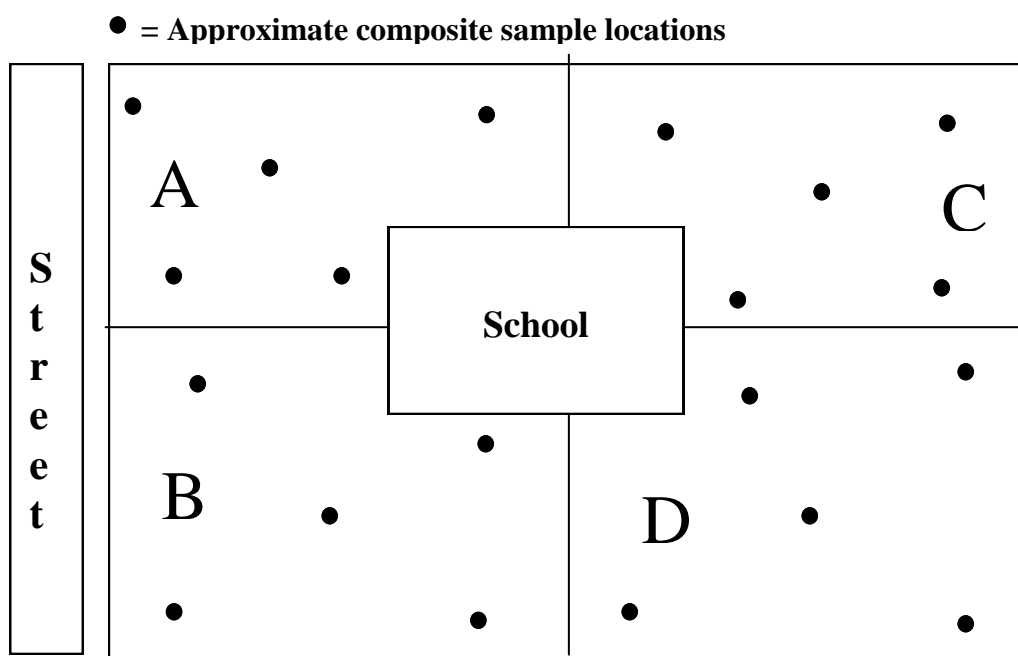
A4.1.1.2 Vacant Lots

All vacant lots will be divided into two halves to correspond with front and back yards. Four 5-point composites will be collected from each half, and each composite sample will be screened using XRF. One 5-point composite will be collected from each half at 0-6 inches, 6-12 inches, 12-18 inches, and 18-24 inches bgs, in an X-shaped pattern, with one sample from each end point of the X and one sample from the center for a total of 8 samples from the vacant lot. If it appears that children are using the area for a play area, a grab sample will be collected from 0-6 inches, 6-12 inches, 12-18 inches, and 18-24 inches bgs at each play area location.



A4.1.1.3 *Schools*

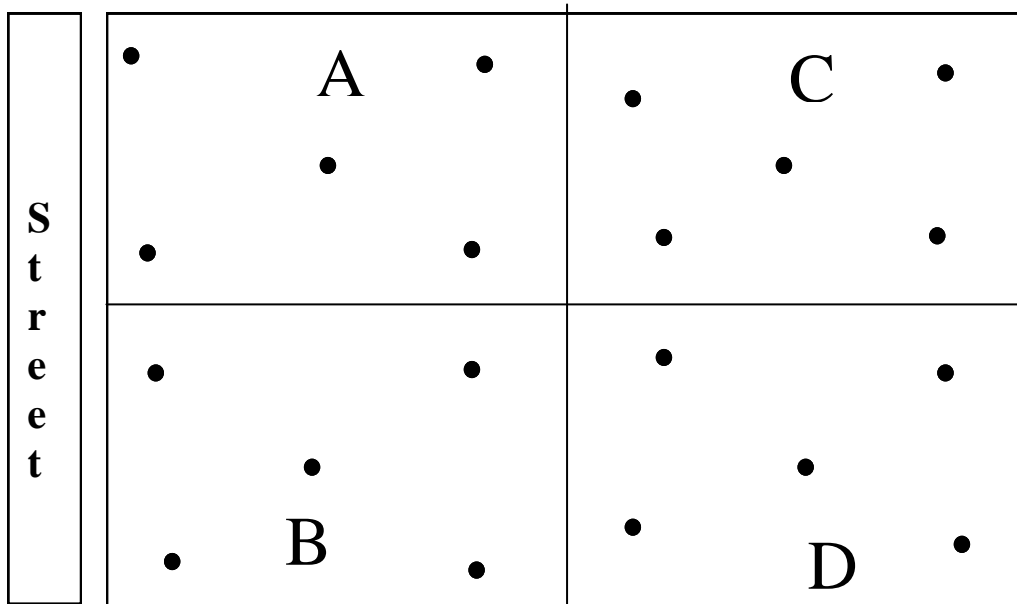
Schools will be sampled by dividing the property into four quadrants. One 5-point composite will be collected from each quadrant at 0-6 inches, 6-12 inches, 12-18 inches and 18-24 inches bgs, in an X-shaped pattern with one sample from each end point of the X and one sample from the center for a total of 16 samples from a school. One grab sample will be collected from each play area location (e.g., playgrounds, baseball/softball fields, soccer fields, and other play areas normally used by schoolchildren at play).



A4.1.1.4 Parks

Parks will be sampled by dividing the property into four quadrants of approximately equal area. Long, narrow parks, such as the park on Kennedy Avenue between 149th and 151st Streets, will be divided into a single row of quadrants. One 5-point composite will be collected from each quadrant at 0-6 inches, 6-12 inches, 12-18 inches, and 18-24 inches bgs, in an X-shaped pattern, with one sample from each end point of the X and one sample from the center. A total of 16 samples will be collected from each park. One additional grab sample will be collected from each play or recreational area location (e.g., playgrounds, baseball/softball fields, soccer fields, and other play/recreational areas normally used by children or adults at play).

● = Approximate composite sample locations



**TABLE A-1
SAMPLING SUMMARY**

Areas	Matrix	Depth (Inches bgs)	No. of Properties	No. of Samples per Property	Total No. of Samples ¹				
					XRF	CLP Metals ^a (20% of XRF samples)	Sieve followed by CLP Metals Analysis ^b (5% of properties)	SVOC, PCB, Pesticides ^c (10% of properties)	VOCs ^c (10% of properties)
Residential Properties and Vacant Lots	Soil	0-6	110	3 ^d	330	66	1	3	0
		6-12		2	220	44	1	1	4
		12-18		2	220	44	1	2	2
		18-24		2	220	44	0	1	1
Gardens and Play Areas	Soil	0-6	22 ^e	1	22	6	1	2	2
		6-12		1	22	6	0	1	1
		12-18		1	22	6	0	0	0
		18-24		1	22	6	0	0	0
Schools	Soil	0-6	1	6 ^f	6	2	1	1	0
		6-12		6	6	2	0	0	1
		12-18		6	6	2	0	0	0
		18-24		6	6	2	0	0	0
Parks	Soil	0-6	4	8 ^g	32	4	1	1	1
		6-12		8	32	4	0	0	0
		12-18		8	32	4	0	0	0
		18-24		8	32	4	0	0	0
TOTAL			115		1,230	246	6	12	12

Notes:

bgs Below ground surface

CLP Contract Laboratory Program

PCB Polychlorinated biphenyl

SVOC

VOC

XRF

Semivolatile organic compound

Volatile organic compound

X-rayfluorescence

a A single 5-point composite sample from 20 percent of the properties will be submitted for CLP metals laboratory analysis.

b A single 5-point composite sample from 5 percent of the residential properties will be submitted and for sieve analysis.

c A single 5-point composite sample from 10 percent of the residential properties will be submitted for CLP metals, SVOC, VOC, pesticides, and PCB laboratory analysis.

d Includes one sample from front yard, one sample from back yard, and one from drip line or gutter outfall.

e The total number of samples collected will vary depending on the number of vegetable garden, flower garden, and play areas found on each property. This estimate assumes that 20 percent of the properties will have one play area / garden area.

f Assuming two school play areas.

g Assuming two play areas per park.

SulTRAC will collect approximately 246 soil samples from the 1,230 composite XRF-screened samples for CLP laboratory analysis (see [Table A-1](#)). All 246 samples will be submitted to the CLP laboratory for total metals analysis to create a XRF correlation factor. Twelve of the samples will be sent to the CLP laboratory for volatile organic compounds (VOC), semivolatile organic compounds (SVOC), polychlorinated biphenyls (PCBs), and pesticides analysis. Six samples will be sent to the CLP laboratory for sieve analysis followed by CLP metals analysis of both size fractions.

Samples will be analyzed using appropriate EPA methods for all chemical analysis and ASTM method for the sieve analysis, as identified in [Section A6.0](#) of this FSP. The sieve analysis samples will follow the recommended methodology suggested by EPA's Technical Review Workgroup ([EPA 2000](#)) to sieve the entire weighted sample through an ASTM number 60 sieve; then weigh and analyze both the coarse ($\geq 250 \mu\text{m}$) and fine ($< 250 \mu\text{m}$) fractions of the sample for CLP metals. QC samples (field duplicate, matrix spike [MS], and matrix spike duplicate [MSD] samples) will be collected as described in Section A11.0 of this FSP.

A4.2 PHASE II SITE INVESTIGATION

Phase II activities will be performed after Phase I activities have been completed and the results have been evaluated. The results from the Phase I remedial investigation will be used to identify data gaps requiring further sampling. This FSP will be amended to reflect sampling during Phase II of the investigation.

A5.0 FIELD SAMPLING PROCEDURES

This section describes the procedures to be used to collect the types of samples described in [Section A4.0](#). Specifically, this section details the procedures and methods that will be used to collect soil screening samples.

As discussed in [Section A4.1.1](#), a soil screening investigation for metals will be conducted on residential properties at the USS Lead site. SulTRAC will collect samples from 110 residential properties, four parks, and one school for metals screening using an XRF ([Figure A-3](#)).

Soil composition at each soil screening locations will be documented in the field notebook. The following information will be recorded for each soil screening location: location number, date completed, time, field personnel's initials, and location sketch with a north directional arrow (with adequate information to locate the individual locations for each component of the 5-point composite sample). The lithologic description will also be recorded for every location and must include color, texture, and lithology. If slag or stamp sands are encountered, this information will be clearly identified in the field notebook. All soil screening sample identification (ID) numbers ([Section A8.2](#)) will be entered in the field log book. All composite soil-screening locations will be photographed with the sample ID number written on a whiteboard and a geographic landmark of some kind in the field of view. Photographs will be archived.

Five-point composite samples will be collected from each yard or quadrant of each selected property in the configuration described in [Section 4.1.1](#). Samples will be collected from 0 to 6 inches, 6 to 12 inches, 12 to 18 inches, and 18 to 24 inches bgs, as recommended in the Superfund Lead-Contaminated Residential Sites Handbook ([EPA 2003](#)). No samples deeper than 24 inches bgs will be collected. Samples will be collected with a 6-inch bucket auger, which will be advanced below the ground surface at each location comprising the 5-point composite sample. One aliquot of soil from each depth at each location will be placed in a separate one-gallon Ziploc™ plastic re-sealable bag, with one bag dedicated to each sample depth. Each bag will be thoroughly composited by shaking and stirring, and the bag will be screened with the XRF and the results recorded in a field notebook. Samples for CLP laboratory analysis will then be collected from the same bag. Any excess soil not used for sampling will be used to fill the auger borings. If necessary, auger borings will be filled to the top with potting soil so that no hole remains in the areas sampled.

Use of the XRF method is restricted to personnel trained and knowledgeable in the operation of an XRF instrument. The XRF technologies described in the XRF method use sealed radioisotope sources to irradiate samples with x-rays. For measurement, the soil sample is placed in a plastic bag, positioned in front of the probe window, and measured. The probe window is placed in direct contact with the plastic bag, mainly to preserve the XRF window quality (see [SOP XRF](#) in [Attachment A](#)).

SulTRAC will send samples from 5% of the properties to a CLP laboratory for sieve analysis. The sieve analysis samples will follow the recommended methodology suggested by EPA's Technical Review Workgroup ([EPA 2000](#)) to sieve the entire weighted sample through an ASTM number 60 sieve, then weigh and analyze both the coarse ($\geq 250 \mu\text{m}$) and fine ($< 250 \mu\text{m}$) fractions of the sample for total CLP metals. SulTRAC will send samples from 10% of the properties to a CLP laboratory for VOC, SVOC, total metals, PCB, and pesticide analysis. The samples submitted for VOC analysis will be collected from deeper than 6 inches bgs due to potential for VOCs to volatilize from shallow soils. Samples for sieve analysis and full-scan samples will be selected to represent the different types of fill material that may be present at the site.

Twenty percent of the XRF screening samples will be sent to a CLP laboratory for total metals analysis to determine the XRF correlation factor. Samples sent for CLP metals analysis will be selected from within three different XRF screening ranges to ensure the accuracy of the correlation factor. The three ranges are shown below. The soil samples shipped to the CLP laboratory will be as equally divided within the three ranges as is possible, to help ensure both the accuracy and the precision of the correlation factor. For example if 15 soil samples are collected, 5 samples from the 0 to 300 ppm range, 5 samples from the 300 to 600 ppm range, and 5 samples from the greater than 600 ppm range will be shipped.

- 0 ppm up to 300 ppm
- 300 ppm up to 600 ppm
- Greater than 600 ppm

All samples will be immediately placed in an iced sample cooler and maintained at a temperature of $4 \pm 2 \text{ }^{\circ}\text{C}$ without freezing until delivery to the laboratory under standard chain-of-custody (COC) protocol.

A6.0 LABORATORY ANALYTICAL METHODS

Table A-2 lists the laboratory analytical methods for the samples collected by SulTRAC. Field investigation samples will be analyzed by the CLP laboratory and Central Regional Laboratory (CRL).

**TABLE A-2
ANALYTICAL METHODS SUMMARY**

Parameter	Analytical Method ^a
SOIL	
VOCs	CLP SOW SOM01.2
SVOCs	CLP SOW SOM01.2
PCBs	CLP SOW SOM01.2
Pesticides	CLP SOW SOM01.2
TAL metals (including mercury)	CLP SOW ILM05.4
Sieve Analysis	CLP SOW ILM05.4 MA

Notes:

CLP Contract Laboratory Program
MA Modified Analysis
PCB Polychlorinated biphenyl
SVOC Semivolatile organic compound
SOW Statement of work
TAL Target Analyte List
VOC Volatile organic compound

a EPA 2005, 2006, and 2008b through f, and 2009; see [Section A12.0](#)

A7.0 DECONTAMINATION PROCEDURES

This section discusses decontamination procedures. The equipment will be decontaminated following the general practices detailed in [SOP 002](#). Disposable sampling equipment will be used to collect individual grab samples only, and will not be decontaminated. Soil samples will be collected with hand augers. The augers will be washed with a brush and non-phosphate detergent (such as Alconox), then washed and thoroughly rinsed with potable water. To prevent cross contamination, measuring and sampling equipment will be decontaminated prior to the initiation of sample collection activities and between each consecutive sampling location.

A8.0 SAMPLE HANDLING PROCEDURES

SulTRAC will collect soil samples; prepare the samples for shipment; complete all necessary documentation; and decontaminate non-disposable equipment. The following sections discuss sample containers, preservatives, and holding times; sample ID; sample documentation; sample COC; and sample packing and shipment.

A8.1 SAMPLE CONTAINERS, PRESERVATION, AND HOLDING TIMES

SulTRAC anticipates collecting soil samples. Sample handling and procedures are different for each type of chemical group analysis and matrix type. [Table A-3](#) summarizes sample container, preservation requirements, and holding-time requirements for this project.

TABLE A-3
SAMPLE CONTAINERS, PRESERVATION, AND HOLDING TIMES

Matrix	Analyte	Sample Container	Preservation Requirements	Maximum Holding Time ^a
Soil	VOCs	Three 40-milliliter (mL) glass containers with polytetrafluoroethylene (PTFE)-lined septa and open-top screw caps, pre-weighted and containing magnetic stir bars, and one sample container with no headspace for moisture content	Cool to 4 ± 2 °C immediately after collection	48 hours to preservation at laboratory/ 14 days for analysis after preservation
Soil	SVOCs	Two 4- or one 8-ounce wide-mouth glass jar(s)	Cool to 4 ± 2 °C immediately after collection	14 days/40 days
Soil	PCBs	Two 4- or one 8-ounce wide-mouth glass jar(s)	Cool to 4 ± 2 °C immediately after collection	14 days/30 days
Soil	Pesticides	Two 4- or one 8-ounce wide-mouth glass jar(s)	Cool to 4 ± 2 °C immediately after collection	14 days
Soil	TAL Metals (including Hg)	Two 4- or one 8-ounce wide-mouth glass jar(s)	Cool to 4 ± 2 °C immediately after collection	6 months (metals and Hg)
Soil	Sieve Analysis	1-Liter wide-mouth glass jar	NA	NA

Notes:

°C Degree Celsius

Hg Mercury

NA Not applicable

PCB Polychlorinated biphenyl

PTFE Polytetrafluoroethylene

SVOC Semivolatile organic compound

VOC Volatile organic compound

a Holding time is measured from the time of sample collection to the time of sample extraction and analysis (EPA 2004)

A8.2 SAMPLE IDENTIFICATION

Samples will be identified using a unique sample ID number. The identifier will have the following format:

Street – sequential number – yard/quadrant – depth – sample type

Sample identifiers will consist of the first three letters or numbers of a street name (e.g., DRU for Drummond, 151 for 151st Street); a sequential number will follow (e.g., “001” for the first sample collected); a yard or quadrant designator (“F” for front yard facing street, “B” for back yard, and “A, B, C, or D” for quadrants); a depth designator (“0 – 6” for zero to 6 inches bgs); and a suffix designating sample type (“D” for duplicate sample, “V” for vegetable garden, “F” for flower garden, “P” for play area sample, and “R” for equipment rinsate). For example, a sample collected from 12 to 18 inches bgs from a play area in the back yard at 4856 Drummond Street which is the 231st sample collected by the sample team would be designated as DRU231-B-12-18-P. A duplicate sample collected from 18 to 24 inches bgs in quadrant C at Carrie Gosh School, located at 455 E 148th Street, which is the 119th sample collected by the sample team, would be designated 148119-C-18-24-D. The sample date and time will be recorded in field notebooks and on chains-of-custody forms. Sample team 1 will begin sequential numbering at “001” and sampling team 2 will begin their sequential numbering at “500” to prevent duplicating sample numbers.

CLP Forms-II Lite™ software will also assign each laboratory sample an ID number. Forms-II Lite was developed to expedite sample documentation, track samples from the field to the laboratory, and reduce the most common documentation issues associated with sampling. Before or during the sampling event, the user will enter information regarding the site, project, sampling team, analysis, location, matrix, collection time and date, and sample and tag numbers.

A8.3 SAMPLE LABELS

Forms-II Lite generates labels for all samples. A sample label will be affixed to each sample container. The label will be completed with the following information:

- Project number
- CLP case number

- CLP sample number
- Sample station name (sample ID number)
- Sample collection date and time
- Preservative
- Sample collector's initials
- Analysis required
- Sample tag number

After labeling, each sample will be preserved as required (see [Table A-3](#)).

A8.4 SAMPLE DOCUMENTATION

Sampling activities will be documented in a field notebook using an ink pen. At the start of each day, the following information will be noted: weather, site conditions, field staff present, subcontractors present, and any conducted safety or other meeting. The field team will record the following information in the field logbook for every sample: collection time, sample ID number (not CLP ID number), sampling depth (if appropriate), sampling location description, field observations, sampler's name, time of sample collection, and analyses. Every MS/MSD and duplicate sample will be clearly designated in the field notebook. Collection of rinsate samples and preparation of trip blanks will be documented with applicable parameters in the same manner described above.

Each page of the field notebook will be dated and numbered (if appropriate); and each day's notes will be signed by SulTRAC personnel. Any residual space on the last page of each day's log will be crossed out with a single line. Each new sampling day shall begin on a new page in the field notebook. Any corrections made during the same day of sampling will be crossed out with one single line, or the term "backnote" can be inserted to account for missed time.

The field team leader will ensure that all documentation in the field notebook is appropriately recorded and will check this daily. Any corrections or additions can be made on a subsequent page with appropriate documentation, although this approach is not recommended, and corrections or additions are best made the same day as the sampling.

All field notebooks must be kept secure at all times by the field team leader during the field work period. As possible, all field notebooks shall be scanned electronically at high resolution (minimum 300 by 300

dots per inch [dpi]). If, after 1 week of continuous field work, field notes cannot be electronically scanned, high-resolution hard copies must be made and kept secure until electronic scanning can be performed. All completed field notebooks and any hard copies will be stored with the project manager in the Chicago office. Field data records will be maintained in accordance with EPA's "Multi-Media Investigation Manual" ([EPA 1992a](#)) and this FSP.

A8.5 SAMPLE CHAIN OF CUSTODY

SulTRAC will use standard sample COC procedures to maintain and document sample integrity during collection, transportation, storage, and analysis in accordance with the SulTRAC RAC 2 Contract Level QAPP. A sample will be considered in custody if one of the following statements applies:

- It is in a person's physical possession or view.
- It is in a secure area with restricted access.
- It is placed in a container and secured with an official seal so that the sample cannot be reached without breaking the seal.

Forms-II Lite generates and prints COC forms, called traffic reports (a laboratory copy and a region copy). The laboratory copy will be sealed inside the lid of the sample shipment container. COC procedures provide an accurate written record that traces the possession of individual samples from the time of collection in the field to the time of acceptance at the laboratory. One COC record will be generated for each container shipped. The COC record also will be used to document all samples collected and the analyses requested. The following information will be documented on the COC form:

- Project name and number (region copy only)
- CLP or CRL case number
- CLP or CRL sample numbers
- Sample tag numbers
- Sampling location (station ID number)
- Name and signature of sampler
- Destination of samples (laboratory name)
- Sample ID number
- Date and time of collection

- Number and type of containers filled
- Analysis(es) requested
- Preservatives used (if applicable)
- Sample designation (grab or composite)
- Special instructions (for example, laboratory needs to sub-sample oversized material or perform additional homogenization)
- Signatures of all samplers
- Signatures of individuals involved in custody transfer, including the date and time of transfer
- Airbill number (if applicable)
- Project contact and telephone number
- Custody seal number

SulTRAC will follow the procedures in the EPA Region 5 CRL “Superfund Amendments and Reauthorization Act (SARA)/Superfund Sample Handling Manual” (EPA 1989) to complete the documentation listed above.

SulTRAC will appoint one of its field technical staff members to serve as the sample custodian. Upon completion of all required documents, the sample custodian will sign and date the documents and list the time of sample collection. The custodian also will confirm the completeness of all descriptive information on the COC forms, which will be included with each shipping container. Two custody seals will be used: one across the latch of the sample shipment container and the other on the opposite side of the container lid. The lid will be securely taped shut for shipment. The field sample custodian will send the original copies of the COC region copies to the project manager, who in turn will submit these to the Region 5 Sample Management Office (SMO) within 5 working days of the work completed. The sample custodian will also scan and retain copies of all COCs (laboratory and region) for the project files.

A8.6 SAMPLE PACKING AND SHIPMENT

The procedures to ship samples collected during this project are summarized below:

- All sample jars will be individually wrapped with bubble wrap or other packing material and placed in their own individual ziplock-type bags. Each sample will have its CLP ID tag (if needed) accompanying the sample package.

- Ice will be double-bagged in large ziplock-type bags and placed at the bottom of the shipping container. If the shipping container has a drain, the drain will be taped shut both inside and outside the shipping container.
- The shipping container will be lined with bubble wrap or other packing material, and all individually packaged samples will be placed into one large plastic bag and tied shut. Sufficient packing material will be used to prevent sample containers from breaking during shipment.
- Additional double-bagged ice will be added on top of the tied plastic bag full of samples. Enough ice will be added to maintain a sample temperature of 4 ± 2 °C. SulTRAC shall prepare, label, and place a temperature blank in each shipping container. SulTRAC shall also include one trip blank in each shipping container.
- If a sampler suspects that any sample contains anomalously high or low concentrations or requires laboratory personnel to take safety precautions, this information will be handwritten directly on the laboratory copy of the COC form.
- The COC form specific to each shipping container will be sealed inside a plastic bag and taped to the inside of the shipping container lid. The COC must be signed by all samplers and the custody seal numbers included on the COC form. A return prepaid airbill will be included with the COC form so the sample shipping container can be returned to SulTRAC.
- The shipping container will be closed and taped shut with strapping tape around both ends.
- Signed and dated custody seals will be placed on the front and side of each shipping container. Wide clear tape will be placed over the seals to prevent accidental tearing.
- The airbill, if required, will be completed before the samples are relinquished to the carrier.
- The COC form will be transported within the taped and sealed shipping container. When the shipping container is received at the analytical laboratory, laboratory personnel will open the shipping container and sign the COC form to document transfer of the samples.
- The Superfund SMO will be notified if the laboratory expects to receive samples on Saturday. SulTRAC will call its CLP sample coordinator, who in turn will notify the SMO.

All shipping containers will be labeled as required by the U.S. Department of Transportation (DOT).

After packing, the samples will be shipped to the CRL or CLP laboratory specified by the EPA Region 5 Regional Sample Control Coordinator.

A9.0 DISPOSAL OF INVESTIGATION-DERIVED WASTE

Investigation-derived waste (IDW) is expected to be minimal. Composite sample soil that is not submitted to a laboratory will be placed back into the augered holes. Equipment will be decontaminated at each yard of each property, and rinsate will be discarded on the ground surface in the yard from which the samples were collected.

A10.0 HEALTH AND SAFETY PROCEDURES

All field activities will be conducted in accordance with the SulTRAC health and safety plan (HASP) ([SulTRAC 2009c](#)). Prior to initiation of field activities, all SulTRAC field personnel and subcontractors will read and sign the HASP, indicating that they understand the HASP and agree to operate in accordance with its requirements. All SulTRAC personnel and subcontractors must have 40-hour hazardous waste and emergency response training, and proof of certification must be filed with the signed HASP. At least one on-site person will have First Aid and cardiopulmonary resuscitation (CPR) training. A complete copy of the site-specific plans, including the updated HASP, will be maintained by the field sampling team at the site.

A11.0 QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

All QA activities will be conducted in accordance with the SAP. A copy of the SAP will be maintained by the field sampling team for immediate use in resolving any QA issues that might arise during field activities.

QC samples will be collected at the following frequencies:

- **Field Duplicate:** One per 10 environmental samples collected, with a minimum of one per sample matrix
- **Trip Blank Samples:** One in each container containing samples for VOC analysis
- **MS/MSD Samples:** One per 20 environmental samples per matrix
- **Rinsate:** One per sample medium per team per week of field work

Field duplicate samples consist of two separate samples collected from the same sampling location and depth using the same equipment and sampling procedures. Duplicate samples will be labeled with the sample type and the suffix “D” added to the sample ID. Equipment rinsate samples will be collected once per week, and will be labeled with the suffix “R”. Because organic data are being collected for informational purposes only and no regulatory decisions will be made based on organic data, no rinsate samples will be collected for organics. A trip blank is a clean sample of a matrix taken from the laboratory (if possible) to the sampling site and transported back to the laboratory, without having been exposed to sampling procedures (typically analyzed only for VOCs). This sample is not to be labeled or identified as a trip blank for the CLP laboratory. Trip blank samples will be labeled with the suffix “T.” The MS/MSD is an environmental sample divided into two separate aliquots, each of which is spiked with known concentrations of target analytes. The two spiked aliquots, in addition to an unspiked sample aliquot, are analyzed separately and the results are compared to determine the effects of the matrix on the precision and accuracy of the analysis. For solid matrices, the MS/MSD does not require extra sample volume collection. All MS/MSD samples will be identified for the CRL and CLP laboratory.

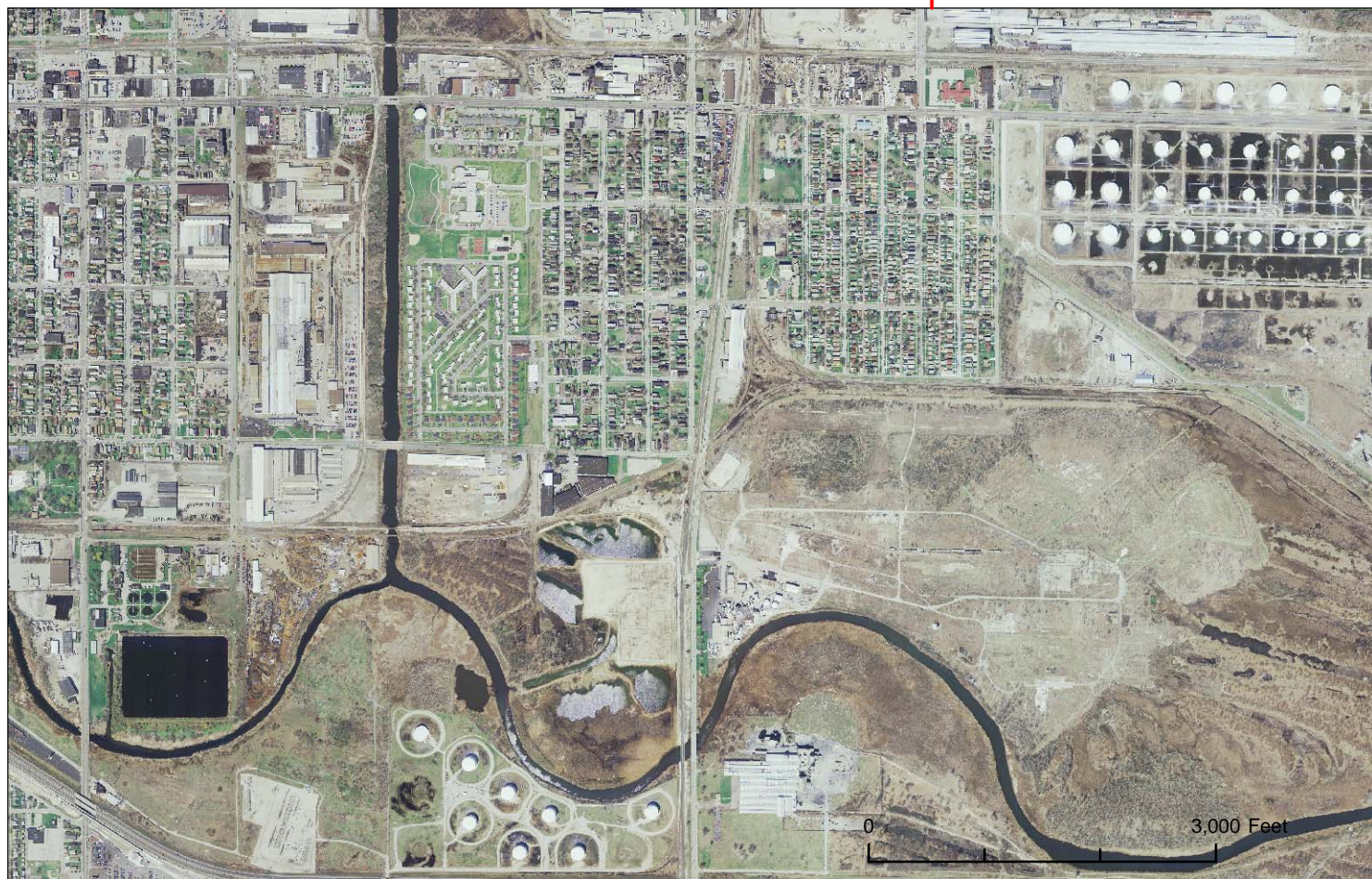
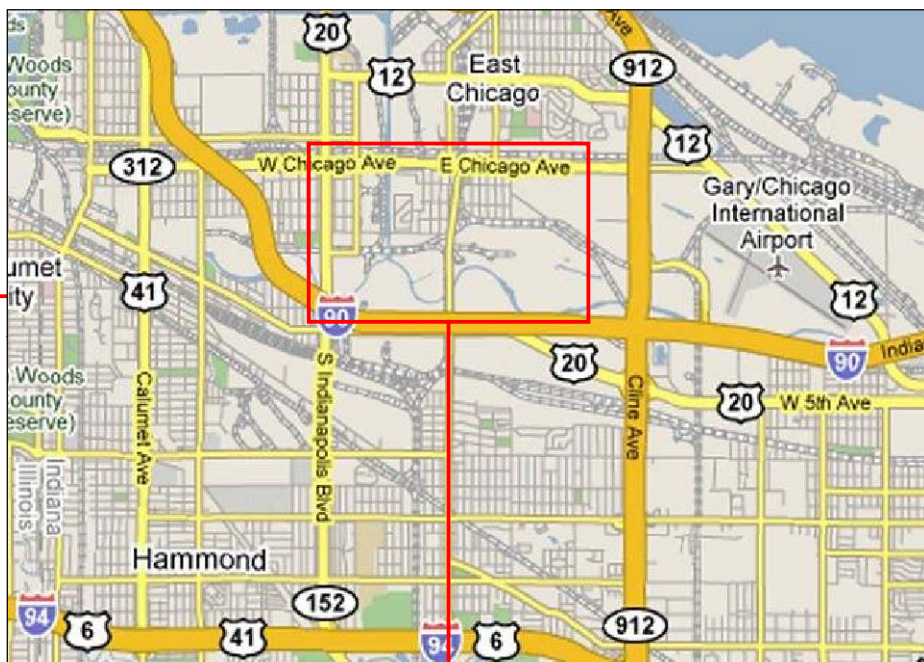
A12.0 REFERENCES

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FIGURES

(Three Sheets)

- **FIGURE A-1, USS LEAD SITE OVERVIEW**
- **FIGURE A-2, USS LEAD SITE STUDY AREAS**
- **FIGURE A-3, USS LEAD NEIGHBORHOOD AREAS**



US SMELTER & LEAD REFINERY
LAKE COUNTY, EAST CHICAGO, INDIANA

FIELD SAMPLING PLAN

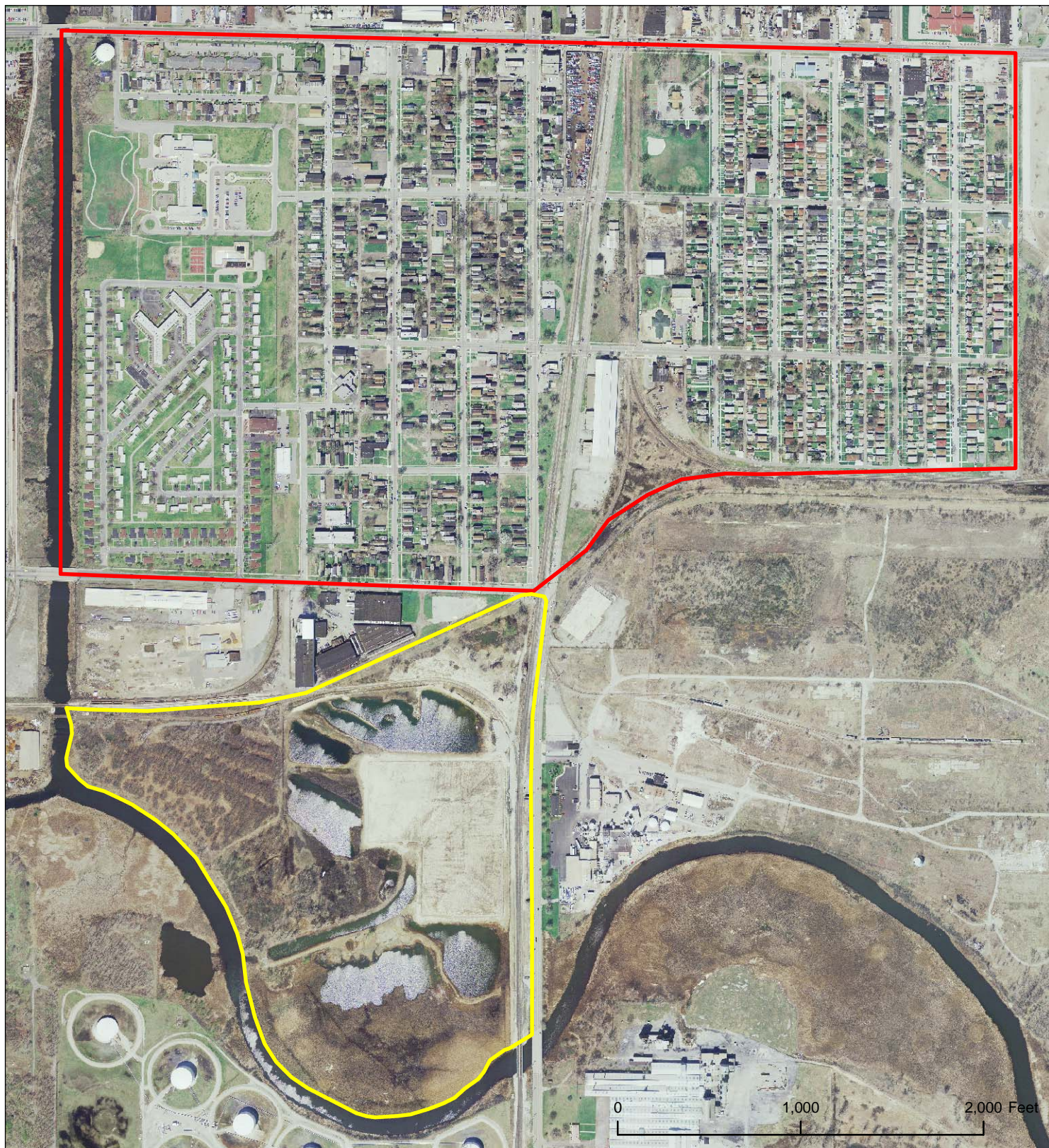
FIGURE A-1

USS LEAD SITE OVERVIEW

Imagery sources (clockwise from upper-left):
ESRI Resource Center
Google Maps
ISDP (Indiana Spatial Data Portal)

EPA REGION 5 RAC 2 | REVISION 0 | OCTOBER 2009





Legend



Residential Area Boundary



Wetlands Study Area Boundary

Imagery source:
ISDP (Indiana Spatial Data Portal)



US SMELTER & LEAD REFINERY
LAKE COUNTY, EAST CHICAGO, INDIANA

FIELD SAMPLING PLAN

FIGURE A-2

USS LEAD SITE STUDY AREAS

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FIGURE A-3: USS LEAD NEIGHBORHOOD AREAS

HAS BEEN REDACTED – ONE PAGE

CONTAINS POTENTIAL PERSONALLY-IDENTIFYING INFORMATION

ATTACHMENT A
STANDARD OPERATING PROCEDURES

SOP 002	GENERAL EQUIPMENT DECONTAMINATION, REVISION NO. 2, DECEMBER 1999
SOP 005	SOIL SAMPLING, REVISION NO. 1, DECEMBER 1999
SOP XRF	EPA METHOD 6200: X-RAY FLUORESCENCE SPECTROMETRY FOR THE DETERMINATION OF ELEMENTAL CONCENTRATIONS IN SOIL, REVISION NO. 3, FEBRUARY 20

SOP APPROVAL FORM

TETRA TECH EM INC.

ENVIRONMENTAL STANDARD OPERATING PROCEDURE

GENERAL EQUIPMENT DECONTAMINATION

SOP NO. 002

REVISION NO. 3

Last Reviewed: June 2009



Quality Assurance Approved

6-19-09

Date

1.0 BACKGROUND

All nondisposable field equipment must be decontaminated before and after each use at each sampling location to obtain representative samples and to reduce the possibility of cross-contamination.

1.1 PURPOSE

This standard operating procedure (SOP) establishes the requirements and procedures for decontaminating equipment in the field.

1.2 SCOPE

This SOP applies to decontaminating general nondisposable field equipment. To prevent contamination of samples, all sampling equipment must be thoroughly cleaned prior to each use.

1.3 DEFINITIONS

Alconox: Nonphosphate soap, obtained in powder detergent form and dissolved in water

Liquinox: Nonphosphate soap, obtained in liquid form for mixing with water

1.4 REFERENCES

U.S. Environmental Protection Agency (EPA). 1992a. “Guide to Management of Investigation-Derived Wastes.” Office of Solid Waste and Emergency Response. Washington D.C. EPA 9345.3-03FS. January.

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1.5 REQUIREMENTS AND RESOURCES

The equipment required to conduct decontamination is as follows:

- Scrub brushes
- Large wash tubs or buckets
- Squirt bottles
- Alconox or Liquinox
- Tap water
- Distilled water
- Plastic sheeting
- Aluminum foil
- Methanol or hexane
- Isopropanol (pesticide grade)
- Dilute (0.1 N) nitric acid

2.0 PROCEDURE

The procedures below discuss decontamination of personal protective equipment (PPE), drilling and monitoring well installation equipment, borehole soil sampling equipment, water level measurement equipment, general sampling equipment, and groundwater sampling equipment.

2.1 PERSONAL PROTECTIVE EQUIPMENT DECONTAMINATION

Personnel working in the field are required to follow specific procedures for decontamination prior to leaving the work area so that contamination is not spread off site or to clean areas. All used disposable protective clothing, such as Tyvek coveralls, gloves, and booties, will be containerized for later disposal. Decontamination water will be containerized in 55-gallon drums (refer to Section 3.0).

Personnel decontamination procedures will be as follows:

1. Select an area removed from sampling locations that is both downwind and downgradient. Decontamination must not cause cross-contamination between sampling points.
2. Maintain the same level of personal protection as was used for sampling.

3. Wash neoprene boots (or neoprene boots with disposable booties) with Liquinox or Alconox solution and rinse with clean water. Remove booties and retain boots for subsequent reuse.
4. Wash outer gloves in Liquinox or Alconox solution and rinse in clean water. Remove outer gloves and place into plastic bag for disposal.
5. Remove Tyvek or coveralls. Containerize Tyvek for disposal and place coveralls in plastic bag for reuse.
6. Remove air purifying respirator (APR), if used, and place the spent filters into a plastic bag for disposal. Filters should be changed daily or sooner depending on use and application. Place respirator into a separate plastic bag after cleaning and disinfecting.
7. Remove disposable gloves and place them in plastic bag for disposal.
8. Thoroughly wash hands and face in clean water and soap.

2.2 DRILLING AND MONITORING WELL INSTALLATION EQUIPMENT DECONTAMINATION

All drilling equipment should be decontaminated at a designated location on site before drilling operations begin, between borings, and at completion of the project. Decontamination may be conducted on a temporary decontamination pad constructed at satellite locations within the site area in support of temporary work areas. The purpose of the decontamination pad is to contain wash waters and potentially contaminated soil generated during decontamination procedures. Decontamination pads may be constructed of concrete, wood, or plastic sheeting, depending on the site-specific needs and plans. Wash waters and contaminated soil generated during decontamination activities should be considered contaminated and thus, should be collected and containerized for proper disposal.

Monitoring well casing, screens, and fittings are assumed to be delivered to the site in a clean condition. However, they should be steam cleaned and placed on polyethylene sheeting on-site prior to placement downhole. The drilling subcontractor will typically furnish the steam cleaner and water.

The drilling auger, bits, drill pipe, any portion of drill rig that is over the borehole, temporary casing, surface casing, and other equipment used in or near the borehole should be decontaminated by the drilling subcontractor as follows:

1. Select an area removed from sampling locations that is both downwind and downgradient. Decontamination must not cause cross-contamination between sampling points.
2. Maintain the same level of personal protection as was used for sampling.
3. Remove loose soil using shovels, scrapers, wire brush, etc.
4. Steam clean or pressure wash to remove all visible dirt.
5. If equipment has directly or indirectly contacted contaminated media and is known or suspected of being contaminated with oil, grease, polynuclear aromatic hydrocarbons (PAH), polychlorinated biphenyls (PCB), or other hard to remove organic materials, rinse equipment with pesticide-grade isopropanol.
6. To the extent possible, allow components to air dry.
7. Wrap or cover equipment in clear plastic until it is time to be used.
8. All wastewater from decontamination procedures should be containerized.

2.3 BOREHOLE SOIL SAMPLING DOWNHOLE EQUIPMENT DECONTAMINATION

All soil sampling downhole equipment should be decontaminated before use and after each sample as follows:

1. Select an area removed from sampling locations that is both downwind and downgradient. Decontamination must not cause cross-contamination between sampling points.
2. Maintain the same level of personal protection as was used for sampling.
3. Prior to sampling, scrub the split-barrel sampler and sampling tools in a wash bucket or tub using a stiff, long bristle brush and Liquinox or Alconox solution.
4. After sampling, steam clean the sampling equipment over the rinsate tub and allow to air dry.
5. Place cleaned equipment in a clean area on plastic sheeting and wrap with aluminum foil.
6. Containerize all water and rinsate; disposable single-use sampling equipment should also be containerized.
7. Decontaminate all equipment placed down the hole as described for drilling equipment.

2.4 WATER LEVEL MEASUREMENT EQUIPMENT DECONTAMINATION

Field personnel should decontaminate the well sounder and interface probe before inserting and after removing them from each well. The following decontamination procedures should be used:

1. Select an area removed from sampling locations that is both downwind and downgradient. Decontamination must not cause cross-contamination between sampling points.
2. Maintain the same level of personal protection as was used for sampling.
3. Wipe the tape and probe with a disposable Alconox- or Liquinox-impregnated cloth or paper towel.
4. If immiscible layers are encountered, the interface probe may require steam cleaning or washing with pesticide-grade isopropanol.
5. Rinse with deionized water.

2.5 GENERAL SAMPLING EQUIPMENT DECONTAMINATION

All nondisposable sampling equipment should be decontaminated using the following procedures:

1. Select an area removed from sampling locations that is both downwind and downgradient. Decontamination must not cause cross-contamination between sampling points.
2. Maintain the same level of personal protection as was used for sampling.
3. To decontaminate a piece of equipment, use an Alconox wash; a tap water wash; a solvent (isopropanol, methanol, or hexane) rinse, if applicable, or dilute (0.1 N) nitric acid rinse, if applicable; a distilled water rinse; and air drying. Use a solvent (isopropanol, methanol, or hexane) rinse for grossly contaminated equipment (for example, equipment that is not readily cleaned by the Alconox wash). The dilute nitric acid rinse may be used if metals are the analyte of concern.
4. Place cleaned equipment in a clean area on plastic sheeting and wrap with aluminum foil.
5. Containerize all water and rinsate.

2.6 GROUNDWATER SAMPLING EQUIPMENT

The following procedures are to be employed for the decontamination of equipment used for groundwater sampling. Decontamination is not necessary when using disposable (single-use) pump tubing or bailers. Bailer and downhole pumps and tubing decontamination procedures are described in the following sections.

2.6.1 Bailers

1. Select an area removed from sampling locations that is both downwind and downgradient. Decontamination must not cause cross-contamination between sampling points.
2. Maintain the same level of personal protection as was used for sampling.
3. Evacuate any purge water in the bailer.
4. Scrub using soap and water and/or steam clean the outside of the bailer.
5. Insert the bailer into a clean container of soapy water. Thoroughly rinse the interior of the bailer with the soapy water. If possible, scrub the inside of the bailer with a scrub brush.
6. Remove the bailer from the container of soapy water.
7. Rinse the interior and exterior of the bailer using tap water.
8. If groundwater contains or is suspected to contain oil, grease, PAH, PCB, or other hard to remove organic materials, rinse equipment with pesticide-grade isopropanol.
9. Rinse the bailer interior and exterior with deionized water to rinse off the tap water and solvent residue, as applicable.
10. Drain residual deionized water to the extent possible.
11. Allow components to air dry.
12. Wrap the bailer in aluminum foil or a clean plastic bag for storage.
13. Containerize the decontamination wash waters for proper disposal.

2.6.2 Downhole Pumps and Tubing

1. Select an area removed from sampling locations that is both downwind and downgradient. Decontamination must not cause cross-contamination between sampling points.
2. Maintain the same level of personal protection as was used for sampling.
3. Evacuate any purge water in the pump and tubing.
4. Scrub using soap and water and/or steam clean the outside of the pump and, if applicable, the pump tubing.
5. Insert the pump and tubing into a clean container of soapy water. Pump/run a sufficient amount of soapy water to flush out any residual well water. After the pump and tubing are flushed, circulate soapy water through the pump and tubing to ensure that the internal components are thoroughly flushed.
6. Remove the pump and tubing from the container.
7. Rinse external pump components using tap water.
8. Insert the pump and tubing into a clean container of tap water. Pump/run a sufficient amount of tap water through the pump to evacuate all of the soapy water (until clear).
9. If groundwater contains or is suspected to contain oil, grease, PAH, PCB, or other hard to remove organic materials, rinse the pump and tubing with pesticide-grade isopropanol.
10. Rinse the pump and tubing with deionized water to flush out the tap water and solvent residue, as applicable.
11. Drain residual deionized water to the extent possible.
12. Allow components to air dry.
13. For submersible bladder pumps, disassemble the pump and wash the internal components with soap and water, rinse with tap water, isopropanol (if necessary), and deionized water, and allow to air dry.
14. Wrap pump and tubing in aluminum foil or a clean plastic bag for storage.
15. Containerize the decontamination wash waters for proper disposal.

3.0 INVESTIGATION-DERIVED WASTE

Investigation-derived waste (IDW) can include disposable single-use PPE and sampling equipment, soil cuttings, and decontamination wash waters and sediments. Requirements for waste storage may differ from one facility to the next. Facility-specific directions for waste storage will be provided in project-specific documents, or separate direction will be provided by the project manager. The following guidelines are provided for general use:

1. Assume that all IDW generated from decontamination activities contains the hazardous chemicals associated with the site unless there are analytical or other data to the contrary. Waste solution volumes could vary from a few gallons to several hundred gallons in cases where large equipment required cleaning.
2. Containerized waste rinse solutions are best stored in 55-gallon drums (or equivalent containers) that can be sealed until ultimate disposal at an approved facility.
3. Label IDW storage containers with the facility name and address, date, contents, company generating the waste, and an emergency contact name and phone number.
4. Temporarily store the IDW in a protected area that provides access to the containers and allows for spill/leak monitoring, sampling of containers, and removal following determination of the disposal method.

SOP APPROVAL FORM

TETRA TECH EM INC.
ENVIRONMENTAL STANDARD OPERATING PROCEDURE

SOIL SAMPLING

SOP NO. 005

REVISION NO. 2

Last Reviewed: June 2009



Quality Assurance Approved

6-19-09

Date

1.0 BACKGROUND

Soil sampling is conducted for three main reasons: for laboratory chemical analysis, laboratory physical analysis, or visual classification and field screening. These three sampling objectives can be achieved separately or in combination with each other. Sampling locations are typically chosen to provide chemical, physical, or visual information in both the horizontal and vertical directions. A sampling and analysis plan is used to outline sampling methods and provide preliminary rationale for sampling locations. Sampling locations may be adjusted in the field based on the screening methods being used and the physical features of the area.

1.1 PURPOSE

Soil sampling is conducted to determine the chemical, physical, and visual characteristics of surface and subsurface soils.

1.2 SCOPE

This standard operating procedure (SOP) describes procedures for soil sampling in different areas using various implements. It includes procedures for test pit, surface soil, and subsurface soil sampling, and describes ten soil sampling devices.

1.3 DEFINITIONS

Hand auger: Instrument attached to the bottom of a length of pipe that has a crossarm or “T” handle at the top. The auger can be closed-spiral or open-spiral.

Bucket auger: A type of auger that consists of a cylindrical bucket 10 to 72 inches in diameter with teeth arranged at the bottom.

Core sampler: Thin-wall cylindrical metal tube with diameter of 0.5 to 3 inches, a tapered nosepiece, a “T” handle to facilitate sampler deployment and retrieval, and a check valve (flutter valve) in the headpiece.

EnCore™ sampler: A disposable volumetric sampling device. It comes in sample sizes of 5 and 25 grams. It is a hermetically sealed, single-use soil sampler made from a high-tech, inert polymer. EnCore™ samplers are used to collect soil samples with zero headspace, as required for volatile organic compound analysis. Each sample is collected using a reusable “T” handle.

Spatulas or Spoons: Stainless steel or disposable instruments for collecting loose unconsolidated material.

Trier: Tube cut in half lengthwise with a sharpened tip that allows for collection of sticky solids or loosening of cohesive soils.

Trowel: Metal or disposable tool with a scooped blade 4 to 8 inches long and 2 to 3 inches wide with a handle.

Split-Spoon (or Split-Barrel) Sampler: Thick-walled steel tube that is split lengthwise. A cutting shoe is attached to the lower end; the upper end contains a check valve and is connected to drill rods.

Thin-Wall Tube Sampler: Steel tube (1 to 3 millimeters thick) with a tapered bottom edge for cutting. The upper end is fastened to a check valve that is attached to drill rods.

Volatile Organics Analysis (VOA) Plunger: Disposable, plastic, single-use soil sample collection device for volatile organic compound sample collection.

1.4 REFERENCES

- U.S. Environmental Protection Agency. (EPA) 1984. “Soil Sampling Quality Assurance Users Guide.” EPA 600/4-84-043.
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<http://www.epa.gov/epawaste/hazard/testmethods/sw846/pdfs/5035.pdf>

1.5 REQUIREMENTS AND RESOURCES

Soil sampling requires the use of one or more of the following types of equipment:

- Spoons and spatulas
- Trowel
- Shovel or spade
- Trier
- Core sampler
- EnCore™ sampler
- VOA Plunger
- Hand auger
- Bucket auger
- Split-spoon
- Thin-wall tube

In addition, the following equipment is also needed for various methods:

- Sample containers, labels, and chain-of-custody forms
- Logbook
- Tape for measuring recovery
- Soil classification information
- Wax or caps for sealing ends of thin-wall tube
- “T” Handles
- Plastic sheeting
- Decontamination equipment
- Drilling equipment
- Backhoe
- Health and safety equipment

2.0 SOIL SAMPLING PROCEDURES

This SOP presents procedures for conducting test pit, surface soil, and subsurface soil sampling. The site sampling plan will specify which of the following procedures will be used.

Soil samples for chemical analysis should be collected in the following order: (1) volatile organics, (2) semivolatile organics, and (3) metals. Once the chemical samples have been containerized, samples for physical analyses can be containerized. Typical physical analyses conducted include (1) grain size distribution, (2) moisture content, (3) saturated permeability, (4) unsaturated permeability, and (5) Atterberg limits. Additionally, visual descriptions of samples, using the Unified Soil Classification System (USCS), should be recorded. Field tests such as head space analyses can also be conducted.

Soil samples for chemical analyses can be collected either as grab samples or composite samples. A grab sample is collected from a discrete location or depth. A composite sample consists of soil combined from more than one discrete location. Typically, composite samples consist of soil obtained from several locations and homogenized in a stainless steel or Teflon[®] pan, tray, or baggie. Refer to the site-specific Quality Assurance Project Plan (QAPP) for methodology for composite sample collection. Samples for volatile organics analysis should not be composited.

All soil samples collected should be packaged and shipped to the laboratories in accordance with SOP 019. All nondedicated or nondisposable equipment used for soil sampling should be decontaminated between sampling locations in accordance with SOP 002.

2.1 SOIL SAMPLE COLLECTION PROCEDURES

Soil samples can be collected as discrete samples for volatile organic compound (VOC) analysis using specialized equipment for preservation in the laboratory or in the field. Soil samples collected for non-VOC analysis can be collected as either grab or composite samples using standard equipment.

2.1.1 Procedure for Preserving and Collecting Soil Samples for VOC analysis

Samples collected for VOC analysis using traditional methods, such as collection in a jar with no preservation, are shown to yield nonrepresentative samples due to loss of VOCs. To prevent such losses,

preservation with methanol or sodium bisulfite may be used to minimize volatilization and biodegradation. This preservation may be performed in the laboratory or in the field, depending on the sample collection methodology used. The specific sampling methodology will be specified in the project-specific QAPP or work plan.

Soil samples to be preserved in the laboratory are collected using SW-846 Method 5035. For samples preserved in the field, laboratories may perform low-level analysis (sodium bisulfate preservation) or high- to medium-level analyses (methanol preservation), depending on the project-specific QAPP.

The following procedures outline the necessary steps for collecting soil samples to be preserved at the laboratory, and for collecting soil samples to be preserved in the field with methanol or sodium bisulfate.

2.1.1.1 Soil Samples to be Preserved at the Laboratory

Soil samples collected for VOC analysis that are to be preserved at the laboratory shall be obtained using a hand-operated, hermetically sealed sample vial such as an EnCore™ sampler. Each sample shall be obtained using a reusable sampling handle (“T” handle) that can be provided with the EnCore™ sampler when requested and purchased. Collect the soil sample in the following manner for each EnCore™ sampler.

The EnCore™ sampler is loaded into the “T” handle with the plunger fully depressed. Press the “T” handle into the soil to be sampled. The plunger will be forced upward as the cavity fills with soil. When the sampler is full, using the “T” handle, rotate the plunger and lock it into place. If the plunger does not lock, then it is not filled with soil. Soft soil may require several plunges or forcing soil against a hard surface such as a decontaminated sample trowel to ensure headspace has been eliminated. Remove soil from the outside of the sampler so a tight seal can be made between the sample cap and the O-ring. With soil slightly piled above the rim of the sampler, force the cap on until the catches hook the side of the sampler. Remove any surface soil from outside of the sampler and place in the foil bag provided with the sampler. Label the bag with sample location information. Typically, collect three EnCore™ samplers per sample location. Decontaminate the “T” handle between sample locations.

Using the EnCore™ sampler eliminates the need for field preservation and the shipping restrictions associated with preservatives. A complete set of instructions is included with each EnCore™ sampler.

After the EnCore™ samples are collected, they should be placed on ice immediately and delivered to the laboratory within 48 hours. The samples must be preserved by the laboratory within 48 hours of collection.

2.1.1.2 Soil Samples to be Preserved in the Field

Soil samples preserved in the field may be prepared for analysis using both the low-level (sodium bisulfate preservation) and high- to medium-level (methanol preservation) methods. If samples effervesce when placed in preservative, it is necessary to collect a sample unpreserved, in deionized water. In addition, an unpreserved sample for determination of moisture content must also be collected when collecting soil samples to be preserved in the field.

Methanol Preservation (High to Medium Level). Bottles may be pre-spiked with methanol in the laboratory or prepared in the field. Soil samples to be preserved in the field with methanol shall utilize 40- to 60-milliliter (mL) glass vials with septum-lined lids. Each sample bottle shall be filled with 25 mL of demonstrated analyte-free purge-and-trap grade 3 methanol. The preferred method for adding methanol to the sample bottle is by removing the lid and using a pipette or scaled syringe to add the methanol directly to the bottle.

Soil shall be collected with the use of a decontaminated (or disposable), small-diameter coring device such as a disposable VOA plunger. The outside diameter of the coring device must be smaller than the inside of the sample bottle neck. To collect the sample, pull the plunger back to the required location, insert it into the soil to be sampled, push the coring device into the soil, extrude the soil sample into the methanol-preserved sample bottle, and cap the bottle tightly. Swirl the sample (do not shake) in the methanol to break up the soil such that all of the soil is covered with methanol. Place the sample on ice immediately.

Sodium Bisulfate Preservation (Low Level). Bottles may be prepared in the laboratory or in the field with sodium bisulfate solution. Samples to be field-preserved using sodium bisulfate are collected using the same procedures described for methanol preservation.

2.1.2 Procedure for Collecting Soil Samples for Non-VOC Analyses

Samples collected for non-VOC analyses may be collected as either grab or composite samples as follows. Using a sampling device, transfer a portion of soil to be sampled to a stainless steel bowl, disposable inert plastic tray, or baggie. Remove roots, vegetation, sticks, and stones larger than the size of pea gravel. Thoroughly mix the soil to obtain as uniform a texture and color as practicable. Transfer the mixed soil to the appropriate sample containers and close the containers. Place the sample containers immediately on ice.

2.2 TEST PIT AND TRENCH SOIL SAMPLING

Test pit and trench soil sampling is conducted when a complete soil profile is required or as a means of locating visually detectable contamination. This type of sampling provides a detailed description of the soil profile and allows for multiple samples to be collected from specific soil horizons. Prior to conducting any test pit or trench excavation with a backhoe, the sampling team should ensure that the sampling area is clear of utility lines, subsurface pipes, and poles.

A test pit or trench is excavated by incrementally removing soil material with a backhoe bucket. The excavated soil is placed on plastic sheeting well away from the edge of the test pit. A test pit should not be excavated to depths greater than 4 feet unless its walls are properly sloped or stabilized. No personnel shall enter any test pit or trench excavation over 4 feet deep; such action would constitute confined space entry and must conform with Occupational Safety and Health Administration (OSHA) regulations at Title 29 of the *Code of Federal Regulations* § 1910.

Personnel entering the test pit may be exposed to toxic or explosive gases and oxygen deficient environments. Air monitoring is required before entering the test pit, and the use of appropriate respiratory gear and protective clothing is mandatory. At least two persons must be present at the test pit before sampling personnel may enter the excavation and begin soil sampling. Refer to project-specific Health and Safety Plans for required safety procedures for excavations.

Soil samples can also be obtained directly from the backhoe bucket or from the excavated material after it has been removed and deposited on plastic sheeting. The sampling personnel shall direct the backhoe excavator to obtain material from the selected depth and location within the excavation. The backhoe

operator shall set the backhoe bucket on the ground in a designated location, at a sufficient distance from the excavation to allow the sampler safe access to the bucket. The backhoe operator shall disengage the controls and signal to the sampler that it is safe to approach the bucket. The soil sample shall then be collected from the center of the backhoe bucket to reduce the potential for cross-contamination of the sample.

Test pits are not practical for sampling at depths greater than 15 feet. If soil samples are required from depths greater than 15 feet, samples should be obtained using test borings instead of test pits. Test pits are also usually limited to a few feet below the water table. In some cases, a pumping system may be required to control the water level within the pits.

Access to open test pits should be restricted by the use of flagging, tape, or fencing. If a fence is used, it should be erected at least 6 feet from the perimeter of the test pit. The test pit should be backfilled as soon as possible after sampling is completed.

Various equipment may be used to collect soil samples from the walls or bottom of a test pit. A hand auger, bucket auger, or core sampler can be used to obtain samples from various depths. A trier, trowel, EnCore™ sampler, VOA plunger, or spoon can be used to obtain samples from the walls or pit bottom surface.

2.3 SURFACE SOIL SAMPLING

The surface soil sampling equipment presented in this SOP is best suited for sampling to depths of 0 to 6 feet below ground surface (bgs). The sample depth, sample analyses, soil type, and soil moisture will also dictate the most suitable sampling equipment. Prior to sample collection, the sampling locations should be cleared of any surface debris such as twigs, rocks, and litter. The following table presents various surface soil sampling equipment and their effective depth ranges, operating means (manual or power), and sample types collected (disturbed or undisturbed).

Sampling Equipment	Effective Depth Range (feet bgs)	Operating Means	Sample Type
Hand Auger	0 to 6	Manual	Disturbed
Bucket Auger	0 to 4	Power	Disturbed
Core Sampler	0 to 4	Manual or Power	Undisturbed
EnCore™ Sampler	Not Applicable	Manual	Disturbed
Spoon/Spatula	0 to 0.5	Manual	Disturbed
Trowel	0 to 1	Manual	Disturbed
VOA Plunger	Not Applicable	Manual	Disturbed

The procedures for using these various types of sampling equipment are discussed below.

2.3.1 Hand Auger

A hand auger equipped with extensions and a “T” handle is used to obtain samples from depths of up to 6 feet bgs. If necessary, a shovel may be used to excavate the topsoil to reach the desired subsoil level. If topsoil is removed, its thickness should be recorded. Samples obtained using a hand auger are disturbed in their collection; determining the exact depth at which samples are obtained is difficult.

The hand auger is screwed into the soil at an angle of 45 to 90 degrees from horizontal. When the entire auger blade has penetrated soil, the auger is removed from the soil by lifting it straight up without turning it, if possible. If the desired sampling depth has not been reached, the soil is removed from the auger and deposited onto plastic sheeting. This procedure is repeated until the desired depth is reached and the soil sample is obtained. The auger is then removed from the boring and the soil sample is collected directly from the auger into an appropriate sample container.

2.3.2 Bucket Auger

A bucket auger, equipped similarly as the hand auger, is used to obtain disturbed samples from depths of up to 4 feet bgs. A bucket auger should be used when sampling stony or dense soil that prohibits the use of a hand-operated core or screw auger. A bucket auger with closed blades is used in soil that cannot generally be penetrated or retrieved by a core sampler.

The bucket auger is rotated while downward pressure is exerted until the bucket is full. The bucket is then removed from the boring, the collected soil is placed on plastic sheeting, and this procedure is repeated until the appropriate depth is reached and a sample is obtained. The bucket is then removed from the boring and the soil sample is transferred from the bucket to an appropriate sample container.

2.3.3 Core Sampler

A hand-operated core sampler (Figure 1), similarly equipped as the hand auger, is used to obtain samples from depths of up to 4 feet bgs in uncompacted soil. The core sampler is capable of retrieving undisturbed soil samples and is appropriate when low concentrations of metals or organics are of concern. The core sampler should be constructed of stainless steel. A polypropylene core sampler is generally not suitable for sampling dense soils or sampling at greater depths.

The core sampler is pressed into the soil at an angle of 45 to 90 degrees from horizontal and is rotated when the desired depth is reached. The core is then removed, and the sample is placed into an appropriate sample container.

2.3.4 Shovel

A shovel may be used to obtain large quantities of soil that are not readily obtained with a trowel. A shovel is used when soil samples from depths of up to 6 feet bgs are to be collected by hand excavation; a tiling spade (sharpshooter) is recommended for excavation and sampling. A standard steel shovel may be used for excavation; either a stainless steel or polypropylene shovel may be used for sampling. Soil excavated from above the desired sampling depth should be stockpiled on plastic sheeting. Soil samples should be collected from the shovel and placed into the sample container using a stainless-steel scoop, plastic spoon, or other appropriate tool.

2.3.5 Trier

A trier (Figure 2) is used to sample soil from depths up to 1 foot bgs. A trier should be made of stainless steel or polypropylene. A chrome-plated steel trier may be suitable when samples are to be analyzed for organics and heavy metal content is not a concern.

Samples are obtained by inserting the trier into soil at an angle of up to 45 degrees from horizontal. The trier is rotated to cut a core and is then pulled from the soil being sampled. The sample is then transferred to an appropriate sample container.

2.3.6 Trowel

A trowel is used to obtain surface soil samples that do not require excavation beyond a depth of 1 foot. A trowel may also be used to collect soil subsamples from profiles exposed in test pits. Use of a trowel is practical when sample volumes of approximately 1 pint (0.5 liter) or less are to be obtained. Excess soil should be placed on plastic sheeting until sampling is completed. A trowel should be made of stainless steel or galvanized steel. It can be purchased from a hardware or garden store. Soil samples to be analyzed for organics should be collected using a stainless steel trowel. Samples may be placed directly from the trowel into sample containers.

2.4 SUBSURFACE SOIL SAMPLING

Subsurface soil sampling is accomplished in conjunction with borehole drilling, for soil sampling from depths greater than approximately 6 feet bgs. Subsurface soil sampling is frequently coupled with exploratory boreholes or monitoring well installation.

Subsurface soil sampling may be conducted using a drilling rig, power auger, or direct-push technology (DPT). Selection of sampling equipment depends upon geologic conditions and the scope of the sampling program. Two types of samplers used with machine-driven augers—the split-spoon sampler and the thin-wall tube sampler—are discussed below. All sampling tools should be cleaned before and after each use in accordance with SOP No. 002 (General Equipment Decontamination). Both the split-spoon sampler and the thin-wall tube sampler can be used to collect undisturbed samples from

unconsolidated soils. The procedures for using the split-spoon and thin-wall tube samplers are presented below.

2.4.1 Split-Spoon Sampler

Split-spoon samplers are available in a variety of types and sizes. Site conditions and project needs, such as large sample volume for multiple analyses, determine the specific type of split-spoon sampler to be used. Figure 3 shows a generic split-spoon sampler.

The split-spoon sampler is advanced into the undisturbed soil beneath the bottom of the casing or borehole using a weighted hammer and a drill rod. The relationship between hammer weight, hammer drop, and number of blows required to advance the split-spoon sampler in 6-inch increments indicates the density or consistency of the subsurface soil. After the split-spoon sampler has been driven to its intended depth, it should be removed carefully to avoid loss of sample material. In noncohesive or saturated soil, a catcher or basket should be used to help retain the sample.

After the split-spoon sampler is removed from the casing, it is detached from the drill rod and opened. If VOA samples are to be collected, EnCore™ samplers or VOA plungers should be filled with soil taken directly from the split-spoon sampler. Samples for other specific chemical analyses should be taken as soon as the VOA sample has been collected. The remainder of the recovered soil can then be used for visual classification of the sample and containerized for physical analysis. The entire sample (except for the top several inches of possibly disturbed material) is retained for analysis or disposal.

2.4.2 Thin-Wall Tube Sampler

A thin-wall tube sampler, sometimes called the Shelby tube (Figure 4), is used to collect soil samples for geophysical analysis. Tube samplers are best suited for collecting cohesive soils such as clays and silts. The tube sampler may be pressed or driven into soil inside a hollow-stem auger flight, wash bore casing, or uncased borehole. The tube sampler is pressed into the soil, without rotation, to the desired depth or until refusal. If the tube cannot be advanced by pushing, it may be necessary to drive it into the soil without rotation using a hammer and drill rod. The tube sampler is then rotated to collect the sample from the soil and removed from the borehole.

After removal of the tube sampler from the drilling equipment, the tube sampler should be inspected for adequate sample recovery. The sampling procedure should be repeated until an adequate soil core is obtained (if sample material can be retained by the tube sampler). The soil core obtained should be documented in the logbook. Any disturbed soil is removed from each end of the tube sampler. If chemical analysis is required, VOA samples must be collected immediately after the tube sampler is withdrawn. EnCore™ samplers or VOA plungers should be filled with soil taken directly from the tube sampler. Before use, and during storage and transport, the tube sampler should be capped with a nonreactive material. For physical sampling parameters, the tube is sealed using plastic caps. The top and bottom of the tube sampler should be labeled and the tube sampler should be stored accordingly.

2.4.3 Direct-Push Technology Sampler

Geoprobe systems utilize DPT. In many cases, DPT is less expensive and faster than collecting soil samples with a standard drilling rig. In addition, the use of DPT causes minimal disturbance to the ground surface and generates little to no soil cuttings. DPT uses acetate or clear polyvinyl chloride (PVC) sleeves for collecting soil samples. Use of the Geoprobe system is described in SOP No. 054.

Upon retrieval of the sampling rod from the ground, the sample sleeve is extruded from the sampling rod. The sleeve is sliced lengthwise twice, to open the sleeve. Soil samples can be collected directly from the opened sleeve. If VOA samples are to be collected, EnCore™ samplers or VOA plungers should be filled with soil taken directly from the opened DPT sampler. Samples for other specific chemical analyses should be taken after the VOA sample has been collected. The remainder of the recovered soil can then be used for visual classification of the sample and containerized for physical analysis. The entire sample is retained for analysis or disposal.

FIGURE 1
HAND-OPERATED CORE SAMPLER

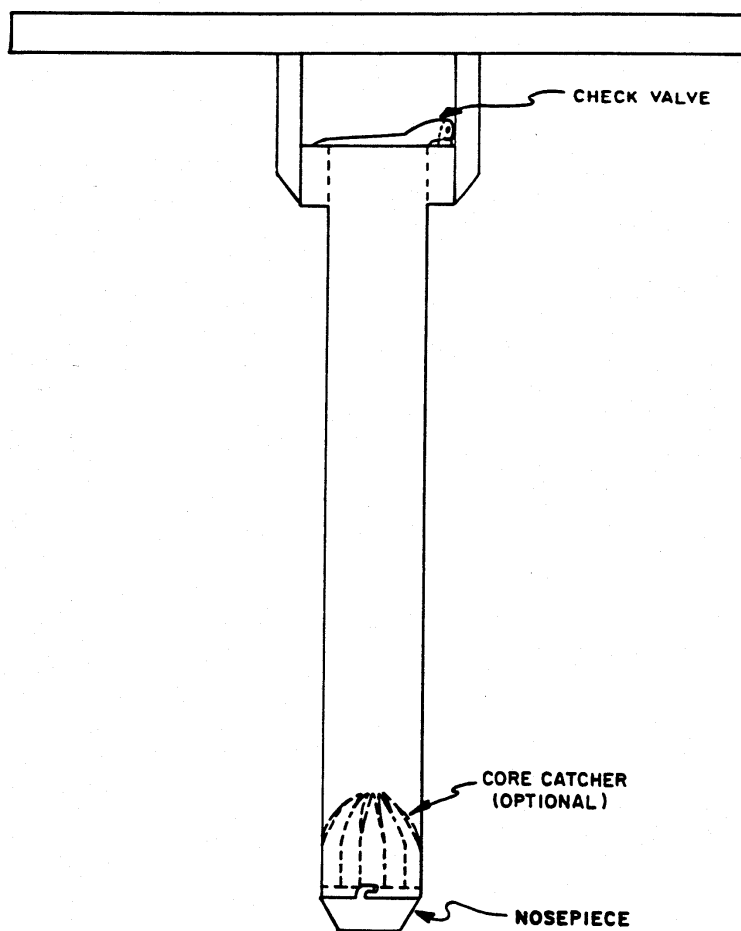


FIGURE 2

TRIER

TRIER

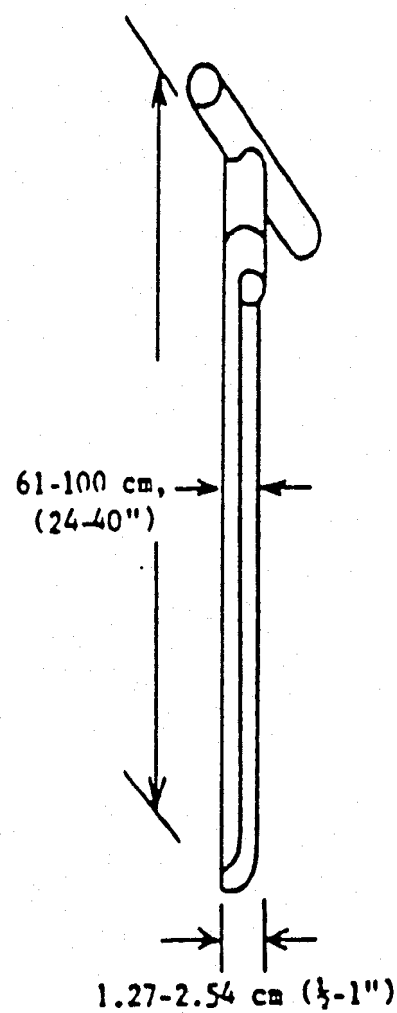


FIGURE 3
GENERIC SPLIT-SPOON SAMPLER

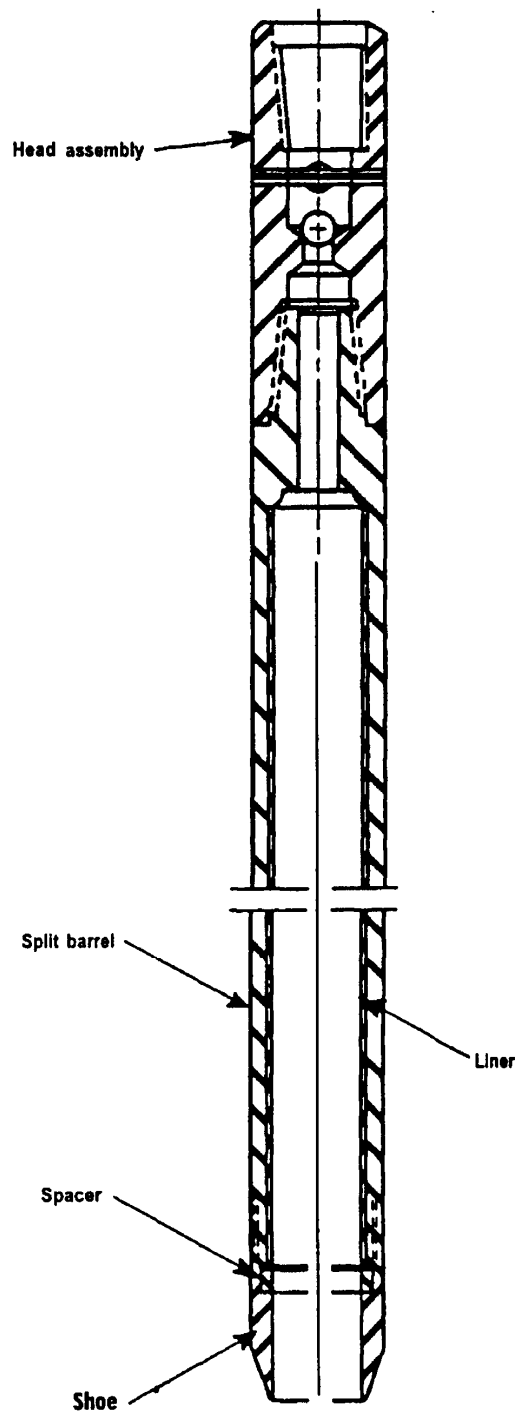
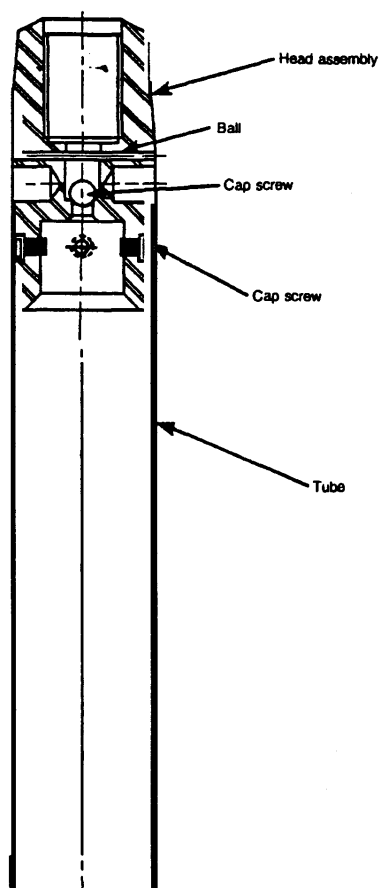


FIGURE 4
THIN-WALL TUBE SAMPLER



California Environmental Protection Agency



SOP MLD 034

**STANDARD OPERATING PROCEDURE FOR THE
DETERMINATION OF ELEMENTAL
CONCENTRATIONS IN AMBIENT AIR BY ENERGY-
DISPERSIVE X-RAY FLUORESCENT (XRF)
SPECTROSCOPY**

Northern Laboratory Branch
Monitoring and Laboratory Division

First Approved Date of SOP: June 17, 1991
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DISCLAIMER: Mention of any trade name or commercial product in this Standard Operating Procedure does not constitute endorsement or recommendation of this product by the Air Resources Board. Specific brand names and instrument descriptions listed in the Standard Operating Procedure are for equipment used by the Air Resources Board laboratory.

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SOP MLD 034

STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF ELEMENTAL CONCENTRATIONS IN AMBIENT AIR BY ENERGY- DISPERSIVE X-RAY FLUORESCENT (XRF) SPECTROSCOPY

1.0 SCOPE

This document describes a non-destructive method for the determination of concentrations of elements in air particulates. All non-volatile elements with atomic numbers from 11 to 92 may be detected by the Air Resources Board's current XRF instrument. The XRF method does not distinguish oxidation states; hence only total elemental concentrations are measured.

2.0 SUMMARY OF METHOD

- 2.1 A sample of air is drawn through thin Teflon[®] membrane filters over a period of twenty-four hours. The sampled volume is about 17 m³ for low-volume total suspended particulate or "total-metal" samples and about 10 m³ for PM_{2.5} samples.
- 2.2 The suspended particles collected by the filters are placed under vacuum in the XRF spectrometer and irradiated with x-rays of various energies. These (primary) x-rays generate fluorescent x-rays, which have characteristic energies for each element in the sample. The fluorescent x-rays, along with primary x-rays scattered from the sample, are individually detected and converted to electrical pulses. These electrical pulses are stored and utilized. Net counts of the characteristic fluorescent x-rays are assumed proportional to elemental surface concentrations (i.e., surface densities in units of µg/cm²). The sample is assumed to be distributed uniformly on the filter.

3.0 SAFETY

- 3.1 The XRF instrument generates X-rays. Exposure to X-rays is injurious to health. The instrument has built-in radiation shielding and safety interlock switches to prevent accidental exposure to X-rays. These safety devices must not be tampered with. Keep the door to the XRF Lab locked when unoccupied and X-rays are being produced.
- 3.2 The X-ray tube exit window and X-ray detector entrance window are made of extremely thin beryllium foil. The windows are extremely fragile and brittle and beryllium is an extremely toxic element. Do not contact the foil

under any circumstances. Use caution when working in the vicinity of the beryllium windows. Open and close the sample chamber cover slowly and carefully. Rapid pressure changes can rupture the windows. If a beryllium window is ruptured, proceed as follows:

- 3.2.1 Do not touch, breathe, or swallow any particles. Do not allow particles to contact skin or clothing.
 - 3.2.2 Using tweezers or the adhesive side of masking tape, collect any broken pieces and particles and place them in a sealed, unbreakable container.
 - 3.2.3 Call a qualified service engineer for additional advice on containment, cleanup, and disposal procedures.
- 3.3 The XRF instrument uses liquid nitrogen (LN) to maintain the detector at low temperatures. The instrument LN dewar must be filled twice per week. LN is a cryogenic hazard and can cause severe burns on contact with skin, eyes, or lungs. Do not allow skin contact with the liquid and do not breathe vapors boiling off the liquid. Wear protective gear (cryogenic gloves, face shield, lab coat) when handling LN. Handle dewars very carefully. LN is also an asphyxiation hazard because nitrogen vapors displace air while boiling off the liquid. Keep the door to the XRF Lab open while handling LN.

4.0 INTERFERENCES

Spectral interferences known as peak overlaps occur when the XRF detector is unable to resolve peaks that are very close to each other. To correct for peak overlaps and obtain accurate peak intensities, an XML peak-fitting method is employed. In this method, a library of pure reference spectra is generated for all elements determined by the method. Then sample spectrum deconvolution is performed by multiple, linear least squares fitting of the reference spectra to the sample spectrum. Additionally, digital filtering of spectra is used to correct for background interferences before the XML treatment is performed. These methods give accurate, net peak, X-ray intensities, which are necessary to accurately determine concentrations of each analyte element.

5.0 EQUIPMENT AND SUPPLIES

5.1 Thermo QuanX-EC XRF Spectrometer with the following configuration.

5.1.1 X-ray Tube: 50 kV, 50 watt, rhodium target, end window emission.

5.1.2 X-ray Generator: Voltage range, 4-50 kV in 1 kV steps. Current range, 0 to 1.98 mA in 0.02 mA steps, 50 watts maximum.

5.1.3 Beam Filters: Automated 8-position beam filter wheel with an open position for direct excitation and 7 transmission filters as indicated in the following table.

Filter Material	Thickness (mm)
Cellulose	-
Aluminum	0.13
Palladium	0.025
Palladium	0.05
Palladium	0.125
Copper	0.377
Copper	0.63

5.1.4 X-ray Detector: LN cooled, high resolution, lithium-drifted silicon, 30 mm² area, 8 µm beryllium window, and 8 liter dewar.

5.1.5 Sample chamber with automated control of air or vacuum environment and the following:

5.1.5.1 Automated 10-position sample tray with sample spinning capability.

5.1.5.2 Sample cups with spinning capability and ability to accommodate either 37 mm or 47 mm diameter filters.

5.1.5.3 Retaining rings, 31 mm I.D. and 47 mm O.D., for holding filters in sample cups.

5.2 Edwards RV 8 vacuum pump for evacuating the spectrometer sample chamber.

5.3 Printer, personal computer system with Wintrace™ XRF applications software.

5.4 Liquid nitrogen pressurized cylinder with 22 psi pressure relief valve.

5.5 NALGENE 4 Liter polyethylene dewar flask for transferring LN.

5.6 Cryogenic gloves, face shield, lab coat.

- 5.7 Pure copper calibration disk for daily energy calibration of detector.
- 5.8 Calibration and quality control standards that are thin vapor-deposited films of elements and compounds purchased from Micromatter Co., Deer Harbor, WA.
- 5.9 High purity reference standards used in peak overlap corrections.

6.0 SAMPLE ANALYSIS

- 6.1 Determine samples that require XRF analysis by running a query in LIMS.
- 6.2 Organize samples by type in batches of ten or less.
- 6.3 Generate a Method Tray List for a sample batch. The XRF computer desktop has shortcuts to method tray lists for the different types of filters.
 - 6.3.1 Double click on the appropriate desktop icon for the type of filters to be analyzed. A Method Tray List appears similar to the figure below.

Method Tray List					
	Sample	Method file	Position	Tray	Count
	PFS Barcode	C:\Documents and Settings\Engineer\My Documents\Methods\PM25 SPECIATION	1	1	1
*					

Ready NUM

- 6.3.2 Click in the cell under "Sample" and enter the first sample ID by reading the barcode on the cover of the sample container.
- 6.3.3 Press <Tab> four times to advance to the cell for the next sample and read its barcode as above.
- 6.3.4 Continue as above until all samples in the batch are entered on the Method Tray List.
- 6.3.5 Following the entry for the last sample in the batch, enter a replicate sample for quality control by typing the ID from the sample in position 1 followed by "A" and pressing <Tab> five times. Make sure that the replicate sample has a 2 in the Tray column.

6.3.6 An example of a properly completed method tray list resembles the figure below.

Method Tray List					
	Sample	Method file	Position	Tray	Count
	PFS01451	C:\Documents and Settings\Engineer\My Documents\Methods\PM25 SPECIATI	1	1	1
	PFS01432	C:\Documents and Settings\Engineer\My Documents\Methods\PM25 SPECIATI	2	1	1
	PFS01440	C:\Documents and Settings\Engineer\My Documents\Methods\PM25 SPECIATI	3	1	1
	PFS01436	C:\Documents and Settings\Engineer\My Documents\Methods\PM25 SPECIATI	4	1	1
	PFS01434	C:\Documents and Settings\Engineer\My Documents\Methods\PM25 SPECIATI	5	1	1
	PFS01435	C:\Documents and Settings\Engineer\My Documents\Methods\PM25 SPECIATI	6	1	1
	PFS01431	C:\Documents and Settings\Engineer\My Documents\Methods\PM25 SPECIATI	7	1	1
	PFS01452	C:\Documents and Settings\Engineer\My Documents\Methods\PM25 SPECIATI	8	1	1
	PFS01439	C:\Documents and Settings\Engineer\My Documents\Methods\PM25 SPECIATI	9	1	1
	PFS01441	C:\Documents and Settings\Engineer\My Documents\Methods\PM25 SPECIATI	10	1	1
	PFS01451A	C:\Documents and Settings\Engineer\My Documents\Methods\PM25 SPECIATI	1	2	1
*					

- 6.4 Load filters into the sample tray according to the positions listed on the method tray list.
 - 6.4.1 Slowly and carefully raise the sample chamber cover.
 - 6.4.2 Remove the cover of a filter container and carefully align an inverted XRF sample holder directly over the filter.
 - 6.4.3 Invert the filter container and the filter should fall into place in the sample holder face-side down.
 - 6.4.4 Place the sample holder with the filter into the appropriate position on the sample tray and place a retaining ring on the filter.
 - 6.4.5 Continue to load samples until entire batch is loaded.
 - 6.4.6 Slowly and carefully lower the sample chamber cover.
- 6.5 Record filter positions in logbook.
- 6.6 Click the Start Acquisition icon to begin analysis.
- 6.7 Spectra is automatically acquired and saved in the method file associated with the filter type. After acquisition is complete for Tray 1, a dialog box prompts to ensure that Tray 2 is in place. Click OK and acquisition continues for a replicate sample.
- 6.8 After acquisition for the replicate sample is complete, an analysis report is displayed in the Acquisition Manager window.

6.9 Close Acquisition Manager and transfer the samples back to containers.

7.0 DATA ANALYSIS

7.1 Data analysis is automatically performed and results are imported into the method file associated with the type of filter analyzed. Further processing of results is necessary prior to transferring data into LIMS.

7.2 Generate a comma separated value (CSV) file for each batch of samples analyzed.

7.2.1 Start the Method Explorer program from the Windows Start Menu and open the method file of the filter type for which data transfer is required.

7.2.2 Expand the "Sample Lists" item in the Method Contents Tree and sample analysis dates and times appear.

7.2.3 Double-click on the appropriate analysis date and "Analysis Results" appears.

7.2.4 Click on Analysis Results and a results table appears. Click on the "Concentration" tab at the bottom of the display. A table appears similar to the following figure.

Sample List: AM-10/05/05 01:06 PM

	Al	Si	P	S	Cl	K	Ca	Ti	
PFS01275	38.445	107.891	6.827	393.278	12.476	37.213	43.325	4.925	
PFS01276	41.847	126.980	0.560	304.300	9.163	69.975	31.030	6.331	
PFS01277	28.009	71.811	0.000	529.637	458.624	76.552	56.784	3.578	
PFS01278	46.414	131.192	2.529	277.712	164.492	53.729	44.378	4.203	
PFS01280	76.181	197.980	4.473	373.262	20.647	84.681	58.094	7.254	
PFS01279	38.166	106.963	0.000	224.550	17.560	65.473	20.932	3.791	
PFS01283	38.277	62.433	0.000	377.189	18.960	42.601	25.496	5.517	
PFS01285	23.375	67.285	1.095	389.558	101.331	35.264	34.389	4.432	
PFS01286	59.365	179.020	2.577	266.903	19.707	70.051	39.290	5.104	
PFS01287	63.280	137.208	2.984	416.023	27.107	54.424	48.859	5.461	
PFS01275A	36.998	106.978	5.454	391.831	20.730	35.863	44.218	4.837	

Peak Intensity Background Intensity Concentrations Normalized Concentrations Uncertainties Chi-Squared Analysts Messages

7.2.5 Click in the cell directly above the first sample ID and the table becomes highlighted in black.

7.2.6 Press <Ctrl+C> to copy the table to the clipboard.

7.2.7 Start Excel and press <Ctrl+V> to paste the table to Excel. Make sure that QC standard results are also copied to the Excel worksheet. A properly completed worksheet resembles the figure below.

	A	B	C	D	E	F	G	H	I	J	K	L	M	N
1	CtIAE327	37.478												
2	CtIFe1131	44.33												
3	CtIPb4475	42.051												
4	CtISn5345	42.519												
5														
6	PFS01273	19.887	56.606	5.235	554.107	34.351	76.071	23.293	4.094	0	0.465	0.698	35.108	0.577
7	PFS01274	50.677	131.681	1.959	378.055	52.481	50.172	46.602	6.034	0.283	0.923	2.46	67.539	0.417
8	PFS01268	175.631	469.028	11.733	192.077	33.897	110.067	219.967	14.093	0.519	1.115	3.111	147.048	1.716
9	PFS01270	72.862	171.073	12.13	280.864	32.175	93.656	66.679	7.968	1.584	0.584	3.1	120.88	1.416
10	PFS01261	17.038	44.01	0	198.076	53.096	28.555	23.476	1.485	0	0.591	1.052	48.632	0.399
11	PFS01273	24.802	55.354	5.061	556.372	28.944	77.148	23.671	2.997	0.59	0.754	0.797	36.998	0.497
12														

7.2.8 Save the Excel file as a *.csv file with the following formats: XPMMDYY.csv for PM2.5 Speciation and XTMMDDYY.csv for Total Metals, where MMDDYY is the month, day, and year the sample batch analysis started.

7.3 Copy the CSV file generated in 7.2.8 to the To_LIMS Folder on the C: drive.

7.4 Log on to IDM LimsLink and start a new worksheet for the appropriate method. Use the CSV file prefix for naming the worksheet.

7.5 Review the results, add replicate and QC samples to LIMS, then send results to LIMS.

8.0 QUALITY CONTROL

8.1 Calibrate the instrument using standards that are thin vapor-deposited films of elements and compounds purchased from Micromatter Co. For most elements, use two standards and a blank. Perform linear regression analysis on intensity (counts per second) verses elemental loading ($\mu\text{g}/\text{cm}^2$) and calculate correlation coefficients. Correlation coefficients must be 0.98 or better for all elements. Complete instrument calibration is

required annually, after instrument repair, or if a control standard result falls outside control limits. Calibrate the instrument using the standards that are listed in the following table:

ID	Element	ug/cm2	ID	Element	ug/cm2
Al 14372	Al	39.8	Ni 4459	Ni	39.0
Al 12891	Al	43.2	CuS 10608	Cu	41.6
SiO 14373	Si	28.6	CuS 10609	Cu	42.7
SiO 10607	Si	31.7	ZnTe 4461	Zn	12.7
GaP 15368	P	15.4	ZnTe 4460	Zn	12.9
CuS 10608	S	13.1	GaAs 15371	As	33.8
CuS 10609	S	14.1	Se 9807	Se	46.4
NaCl 15369	Cl	28.2	Se 9808	Se	47.1
KI 6044	K	11.6	CsBr 15372	Br	17.4
KI 6045	K	12.3	RbI 15373	Rb	19.2
CaF2 15370	Ca	24.0	SrF2 15374	Sr	34.1
Ti 4449	Ti	24.2	YF3 15375	Y	27.7
Ti 9804	Ti	40.3	MoO3 5338	Mo	32.4
V 3312	V	19.6	MoO3 5337	Mo	33.3
V 4451	V	47.8	Sn 9813	Sn	41.8
Cr 3313	Cr	25.0	Sn 9814	Sn	47.4
Cr 4452	Cr	42.7	Sb 5347	Sb	43.4
Mn 4455	Mn	39.1	Sb 5348	Sb	46.2
Mn 4454	Mn	53.9	BaF2 3316	Ba	17.7
Fe 4457	Fe	38.4	BaF2 9816	Ba	36.4
Fe 10613	Fe	51.7	HgAg 15376	Hg	20.6
Co 3509	Co	32.4	Pb 3317	Pb	18.8
Co 5334	Co	48.3	Pb 11315	Pb	49.2
Ni 3315	Ni	20.2	-	-	-

- 8.2 The limit of detection (LOD) for each element must be verified annually. The LOD is calculated using the x-ray counts in a blank spectrum. The counts in a blank spectrum for a given element are divided by the analysis time of the blank spectrum, then multiplied by the time of the given analysis condition. The square root of the resulting number (bkg. cts) is taken and multiplied by three to obtain the number of counts corresponding to the elemental LOD. This number is then converted to $\mu\text{g}/\text{m}^3$ using the appropriate elemental calibration factor C, sample area, sample volume, and analysis time:

$$\text{LOD} = 3(\text{bkg. cts})^{1/2} * \text{C} * (\text{sample area}) / ((\text{m}^3) * (\text{time}))$$

- 8.3 Perform daily energy calibration of the spectrometer to ensure that detected X-rays appear at the correct energy.
 - 8.3.1 Place the copper calibration disk in position 10 of the sample tray.
 - 8.3.2 Start the Acquisition Manager program from the Windows Start Menu.
 - 8.3.3 Click Acquire > Energy Calibration to start the process.
 - 8.3.4 Calibration is performed automatically and an energy calibration report is displayed. The error should be less than 4eV.
 - 8.3.5 Save the report in the Reports folder and close Acquisition Manager.
- 8.4 Analyze control standards before and after every sample batch.
 - 8.4.1 Double click on the QC Standards.mtl desktop icon.
 - 8.4.2 Load the QC standards into the sample tray using the positions listed on the method tray list.
 - 8.4.3 Click the Start Acquisition icon to begin analysis.
 - 8.4.4 After acquisition for the control standards is complete, an analysis report is displayed in the Acquisition Manager window.
 - 8.4.5 Review the control standard results to ensure that they are within the established Control Limits. If a result is not within limits, then the control standard must be reanalyzed. If a second result is also not within limits, analysis must be stopped and the problem investigated.
 - 8.4.6 Close Acquisition Manager.
- 8.5 Analyze at least one replicate sample in every sample batch.

9.0 MAINTENANCE

- 9.1 The XRF detector requires a continuous supply of LN. Fill the instrument LN dewar twice a week. The LN level must be maintained whether or not the system is in use. The dewar is located under the left side access cover of the instrument.

- 9.1.1 Keep the door to the XRF Lab open during the filling process.
- 9.1.2 Wear cryogenic gloves, face shield, and lab coat while transferring LN.
- 9.1.3 Place the 4 Liter dewar flask under the transfer hose of the LN pressurized cylinder. Slowly open the liquid valve on the cylinder and transfer enough LN to fill 4 Liter dewar about $\frac{3}{4}$ full. Place cover on dewar flask.
- 9.1.4 Open access cover on left side of instrument and place cardboard shield over opening to protect detector electronics during LN filling.
- 9.1.5 Remove cap of instrument dewar and place a funnel into the neck of the dewar.
- 9.1.6 Slowly and carefully pour LN into the funnel until the level reaches the bottom of the dewar neck.
- 9.1.7 Wipe condensation off of dewar cap and replace cap on instrument dewar.
- 9.1.8 Record LN fill in logbook. Allow 1 to 3 hours for the detector to stabilize before operating instrument.
- 9.2 Check the vacuum pump oil level monthly. The oil level should be in the middle of the sight glass. If the oil appears dark brown or has a burnt smell, it should be replaced using the same type of oil. It is important to use only pure hydrocarbon oil in the vacuum pump (NO oil containing silicones, which would contaminate the system with silicon).
- 9.3 The QuanX system is covered under a maintenance agreement. For service, repair, or technical support contact Thermo at 1-800-495-3839. Our system identification number is 136247.

10.0 REFERENCES

Thermo Electron Corporation, *QuanX-EC Operator's Manual*, WinTrace Software Version 3.0, Revision A.

Thermo Electron Corporation, *QuanX-EC Technical Manual*, Revision B.

U.S. Environmental Protection Agency, 1999. Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air, Compendium Method IO-3.3, *Determination of Metals in Ambient Particulate Matter Using X-Ray Fluorescence (XRF) Spectroscopy*, EPA/625/R-96/010a, Office of Research and Development.

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